DETERIORATION OF GNEISS DUE TO LIMEWASH
AT EASTERN STATE PENITENTIARY

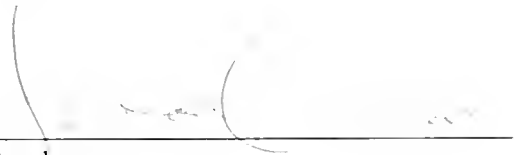Elizabeth Lael Anthony Johansen

A THESIS

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Chapter 1: Introduction

1.1 Building Stone

Of the innumerable materials humankind has used to create shelter in all its forms over the millennia, stone is notable for its universal use, wide variety and general durability. The working properties of stone, its ability to be shaped and joined in a masonry system, have resulted in an extensive patrimony of stone buildings, some thousands of years old.

The body of research in the deterioration and conservation of building stone is extensive. Characteristics of various materials, their deterioration mechanisms, and treatments targeted to specific pathologies have been well researched and documented. As the field of architectural conservation is relatively young and has been pioneered by European practitioners and scholars, the focus of such studies has to date been largely on classes of materials employed most widely in European monumental construction. These materials include sandstones, marbles, limestones and granites. Initial investigations were necessarily targeted to the oldest, most culturally significant sites as they began to manifest rapid deterioration following the onset of the Industrial Revolution after surviving thousands of years of environmental exposure in European and Mediterranean settings. Consequently, the literature devoted to marble and limestone is extensive, as these were the building materials of the Greek and Roman structures central to the

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western cultural heritage. Subsequent studies have generally addressed the problems of

other calcareous stones, sandstones, granites, with some attention to additional specific

stones. However, there are materials for which this research has yet to be undertaken. As

our interest in more recently constructed buildings and structures comprised of more

specific local materials expands, we have the opportunity to contribute to the literature.

The aim of this thesis is to provide one such contribution: a case study devoted to the

deterioration of Wissahickon schist and gneiss.

Two principal deterioration phenomena resulting from the introduction of foreign matter

into building stone are widely researched and published: the contributions of airborne

pollutants and salts, and the effects of waterborne salts. Other mechanisms, including

thermally induced stresses due to insolation, freeze/thaw cycling, seismic activity, and

inappropriate building techniques and materials have also been investigated. The focus of

this study is the interaction of the subject stone with introduced material, rather than the

effects of inherent microstructure or gross construction design on the material's survival.

1.2   Material Selection: Wissahickon Schist and Gneiss

Abundant and easily quarried, the Wissahickon schist and gneiss of southeast

Pennsylvania have been used extensively in all manner of construction since the area was

first settled. From modest residences to extensive institutional and religious structures,

these materials are literally the building blocks of greater Philadelphia. Although the materials are widely used, no literature exists discussing the standard applications and treatments of stone in service, and the problems of weathering and deterioration in situ that commonly arise. The purpose of this thesis is to present one such observed deterioration pattern as a case study, to identify the mechanisms in action, and suggest further investigations that will contribute to the survival of this architecture.

1.3   Site Selection: Eastern State Penitentiary

The investigator's interest in Wissahickon schist and gneiss masonry construction led to the selection of Eastern State Penitentiary as a study site. This large complex of buildings, begun in 1822, was under constant expansion and development until well into the 1960s. The City of Philadelphia certified the prison as an historic property in 1958, and in 1965 the site was given National Historic Landmark status by the federal government. [1] In 1971 the prison was closed, and it remained abandoned until 1991 when stabilization efforts began. The site was included in the World Monuments Watch's 1996-1997 list of the 100 Most Endangered Sites, and efforts continue to forestall deterioration while a financially viable alternate use is sought.

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[1] http://www.libertynet.org/e-state/history.html (February 13, 1999)

Although the material under investigation is observed to display numerous weathering patterns throughout the prison, the site's complexity dictated that the study focus on a small area and a single deterioration context. For this reason, this investigation is limited to the southwest facade of Cell Block Seven, built between 1834 and 1836, the last of John Haviland's buildings.[2] This facade consists of three distinct sections: the central wall, a two-storey gable-end wall that encloses the cell block itself, and the east and west wing walls, which delimit the original exercise yards, now roofed over and converted to interior spaces.

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[2] Norman Johnston, Kenneth Finkel and Jeffrey A. Cohen, *Eastern State Penitentiary: Crucible of Good Intentions* (Philadelphia: Philadelphia Museum of Art, 1994), 43.

Figure 1: Ground Level Overall Plan of Eastern State Penitentiary

Cell Block Seven originates at the central Observation Tower and extends to the southwest.
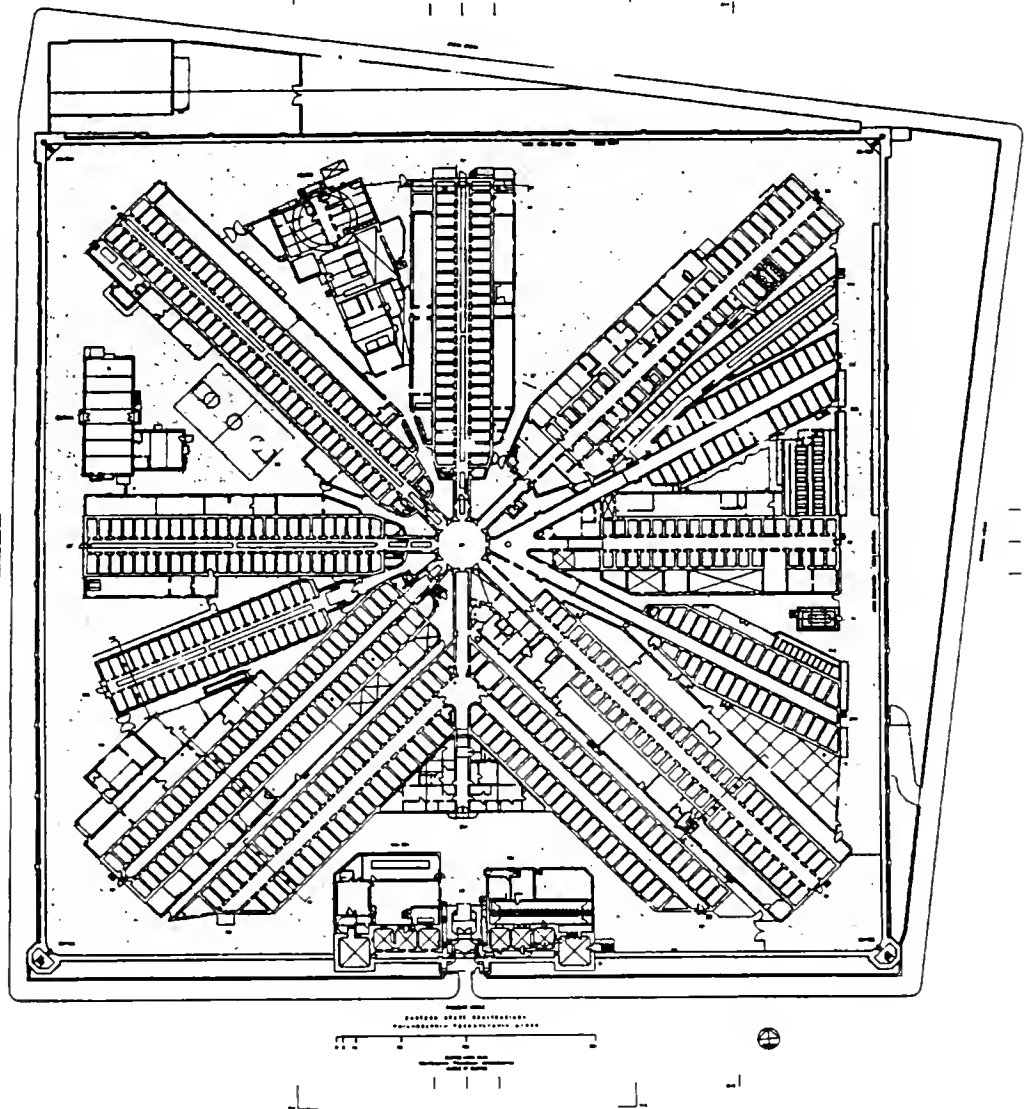
Marianna Thomas Architects, *Eastern State Penitentiary Historic Structures Report.* Philadelphia, 1994. Figure A28. Marianna Thomas Architects, 1994.

Figure 2: Ground Level Southwest Quadrant Plan of Eastern State Penitentiary

Haviland's original buildings are shown in poché. The short southwest wall facing the

perimeter wall corner is the elevation under investigation.
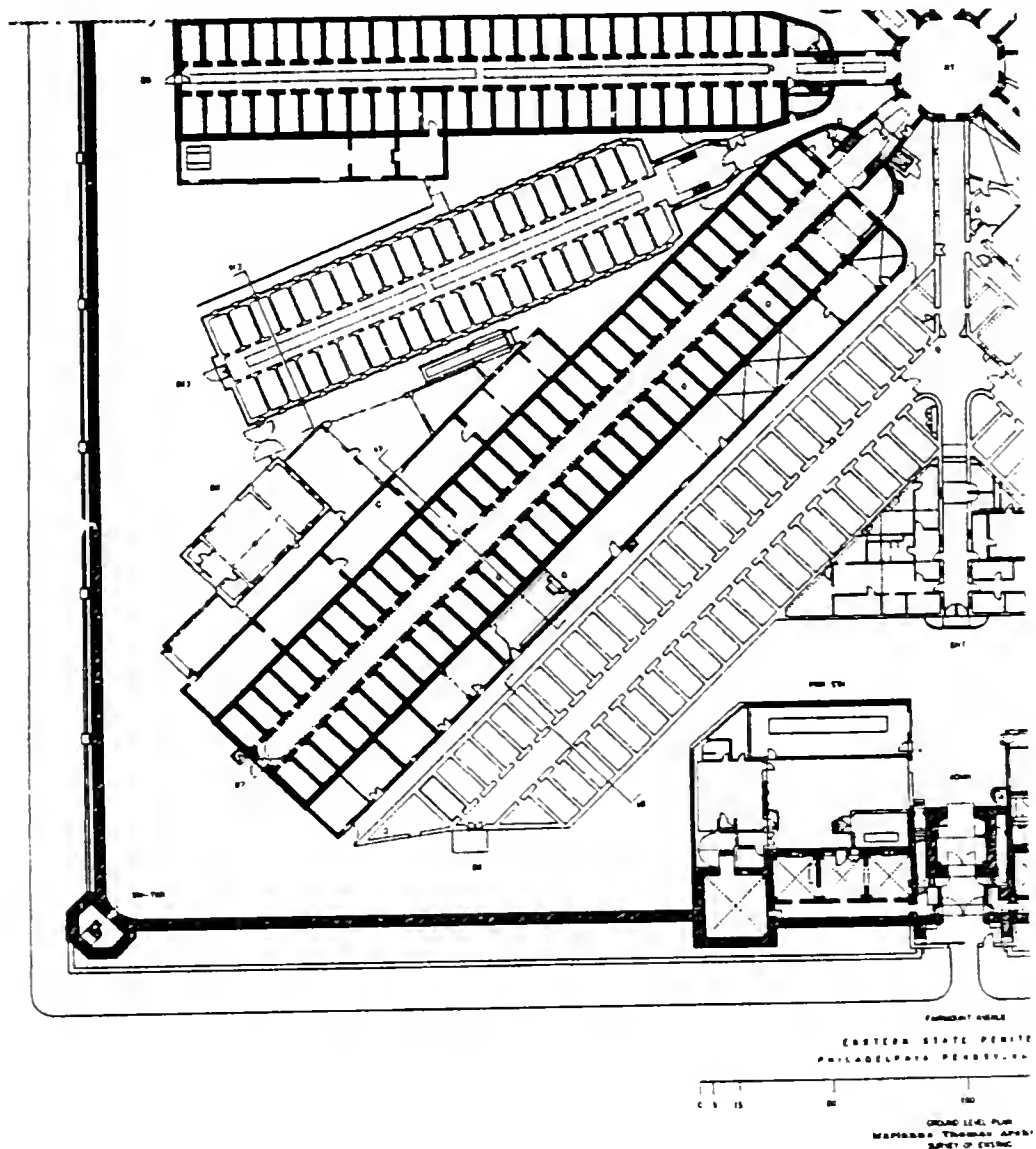
Marianna Thomas Architects. *Eastern State Penitentiary Historic Structures Report*. Philadelphia, 1994. Figure A36. Marianna Thomas Architects, 1994.

Figure 3: Cell Block Seven Facade, Southwest Elevation

Photo: E. Johansen

All three walls are of the same construction: ashlar gneiss facing stones approximately 4"-6" thick with rubble fill.[3] These facing stones are dressed smooth but not polished, and diminish in size as they approach the eave line, producing the visual effect of perspective and exaggerated height.[4] This technique may reflect the relative ease with which smaller stones are set at a greater height. Whether intentional or merely pragmatic, the effect is noteworthy. The masonry units are set in a mud mortar and have undergone occasional repointing.[5] It is likely that more recent repointing campaigns have included Portland cement-based mortar. Some areas within the perimeter walls have been parged with what appears to be Portland cement, with resultant large-scale losses.

Here, as at other locations within the penitentiary perimeter wall, the walls have been limewashed to a height of approximately ten feet. The purpose of this treatment is not known with certainty, although it was likely done to enhance security by rendering hiding in the buildings' shadows nearly impossible. Photographic evidence indicates that at the interior corners of the perimeter walls the limewashed area was used as a backdrop for sports activities in later years,[6] although it is likely that this was an unintended benefit and not the principal impetus for the treatment. Moreover, this use would not

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[3] This information was provided by Sally Elk in conversations between November, 1998 and February, 1999 and confirmed by visual examination on site.

[4] Kieran, Timberlake and Harris, *National Historic Landmark Building Condition Assessment Report: Eastern State Penitentiary* (Philadelphia: unpublished, 1989), Vol. 1. Building Seven, Cell Block Seven, ii.

[5] Marianna Thomas Architects, *Eastern State Penitentiary Historic Structures Report* (Philadelphia: unpublished, 1994) Vol. 1, 89.

[6] Author's conversation with Sean Kelly and Sally Elk, December 1998.

satisfactorily explain the application of limewash on cell block elevations.   Although the exact reason for treating the Cell Block Seven facade in this manner is not known, it is clear that the treatment has resulted in accelerated deterioration of the otherwise relatively durable stone.

Figure 4: Differential Deterioration

Note the relative soundness of the upper material and the deterioration of the lower area.

Photo: E. Johansen

This thesis will investigate the mechanisms contributing to the differentially aggressive deterioration of the limewashed areas of this wall. Through tests of deteriorated material and sound samples, this investigation will attempt to determine the relative contributions of the limewash itself and environmental pollution to the observed deterioration.

Chapter 2: Eastern State Penitentiary Site History

2.1   Historic Summary

Commissioned in 1821, begun in 1822 and completed in 1836, John Haviland's Eastern State Penitentiary stands today as a monument to the progressive social ideals of nineteenth century Quaker Philadelphia and the power of architecture to transform its users and inhabitants. Occupying the block bordered by Fairmount Avenue, Corinthian Avenue, 22nd Street and Brown Street in Philadelphia, Haviland's original seven cell blocks, perimeter wall, observatory and front house still stand, and have been joined over the last century by eight additional cell blocks and numerous ancillary buildings. Although prison operations ceased in 1971, the imposing structure maintains its general form from that time and persists in its role as the model for solitary confinement penitentiary design worldwide.

Haviland's design for Eastern State Penitentiary embodied the convergence of two generations of thought regarding penology reform. The British penal tradition was one of group incarceration and hard labor, with corporal and capital punishment handed down for certain crimes. The Quakers despised such treatment, and in 1776 members of this group joined with other prominent Philadelphians to found the Philadelphia Society for Alleviating the Miseries of Public Prisons, later called the Pennsylvania Prison Society.

Robert Smith's Walnut Street Jail, begun in 1773 and derided after the Revolution by prison reformers as exemplifying all that was wrong with the penal philosophy of the day, became in 1790 a state penitentiary and was enhanced by the addition of "a small cellblock within the prison enclosure to provide solitary confinement for the 'more hardened and atrocious offenders' who were now being given prison terms rather than death sentences."[7] Although by the time it was closed in 1835, replaced by Eastern State Penitentiary, the earlier jail had lost the capacity to accommodate solitary prisoners, the experiment that would come to fruition at Fairmount under Haviland's direction, on the site then commonly known as Cherry Hill, had begun.

Based firmly in the philosophy of solitary confinement for the purpose of inducing reflection and penitence, Eastern State Penitentiary's original form was a hub-and-spoke design, with seven cell blocks radiating from a single observation hub in the center. Haviland hoped to achieve with this approach the unification of "convenience, strength, and economy"[8] and added that the additional benefits of this design were "watching ... and ventilation."[9] From the observation hub, every cell was designed to be visible to the guards. As construction progressed, it became clear that the original 250 cells would not meet the existing need for prison space, consequently cell blocks four, five, six, and seven

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[7] Johnston, Finkel and Cohen, *Crucible*, 26.

[8] John Haviland, "Explanation of a design for a penitentiary," Daybook no. 1, Haviland Papers, Van Pelt Library, University of Pennsylvania.

[9] Ibid.

were built in two storeys rather than the originally intended single storey configuration.[10] The original plan was to provide each prisoner with a cell and an exercise yard. When it became clear that two-storey blocks were required, the challenge arose of providing exercise areas for the upper-storey residents. Haviland handled this problem by providing these inmates two cells, both smaller than those of the first floor, with a communicating door. In addition to moderately sized cells and private work and exercise spaces, Haviland included flush toilets, faucets, and central heat. An opening allowed food and work materials to be passed into the cells from the radial halls, and a ventilation system attempted to reduce illness caused by "miasmas" or confined air.[11] Despite these advanced design features, problems did arise and later adjustments to the mechanical systems were required.[12]

The whole structure was enclosed within four joined perimeter walls, each 42 feet high, 670 feet long, and six feet thick at the base.[13] The corners of the walls are surmounted by observation towers. In addition to the original buildings, the area now holds administration additions (1964), Death Row (1956), cell blocks eight through fourteen (1877-1926), a kitchen (1903), laundry (1905), garage and greenhouse.[14]

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[10] Johnston, Finkel and Cohen, *Crucible*, 41-43.
[11] Ibid., 32.
[12] Ibid., 43.
[13] KTH, *Assessment*, Vol. 1. Building Seventeen, Walls and Corner Towers, i.
[14] Ibid., Vol. 1. Preliminary Construction Chronology.

A number of publications and documents devoted to this site, its role in the history of both Philadelphia and penal philosophy and practice in America and around the world, and its architectural evolution. Chief among these are Johnston's *Crucible of Good Intentions* (1994), Marianna Thomas Architects' *Historic Structures Report*, and Kieran, Timberlake and Harris' *National Historic Landmark Building Condition Assessment Report* (1989).

2.2   Cell Block Seven Construction Chronology

This wing was initially built between 1834 and 1836 in the ashlar-faced rubble construction described above. By 1872 the wing had been extended to accommodate a boiler room and laundry.[15] The plans from this period indicate that the extension was two storeys in height. Figure 4 shows the first floor plan, while Figure 5 indicates the extension's full width at the second floor level, in contrast to the narrow existing cell block structure at that level.

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[15] Marianna Thomas Architects, *Historic Structures Report*, Vol. 1, p.189.

Figure 5: First Floor Plan with Addition, 1900
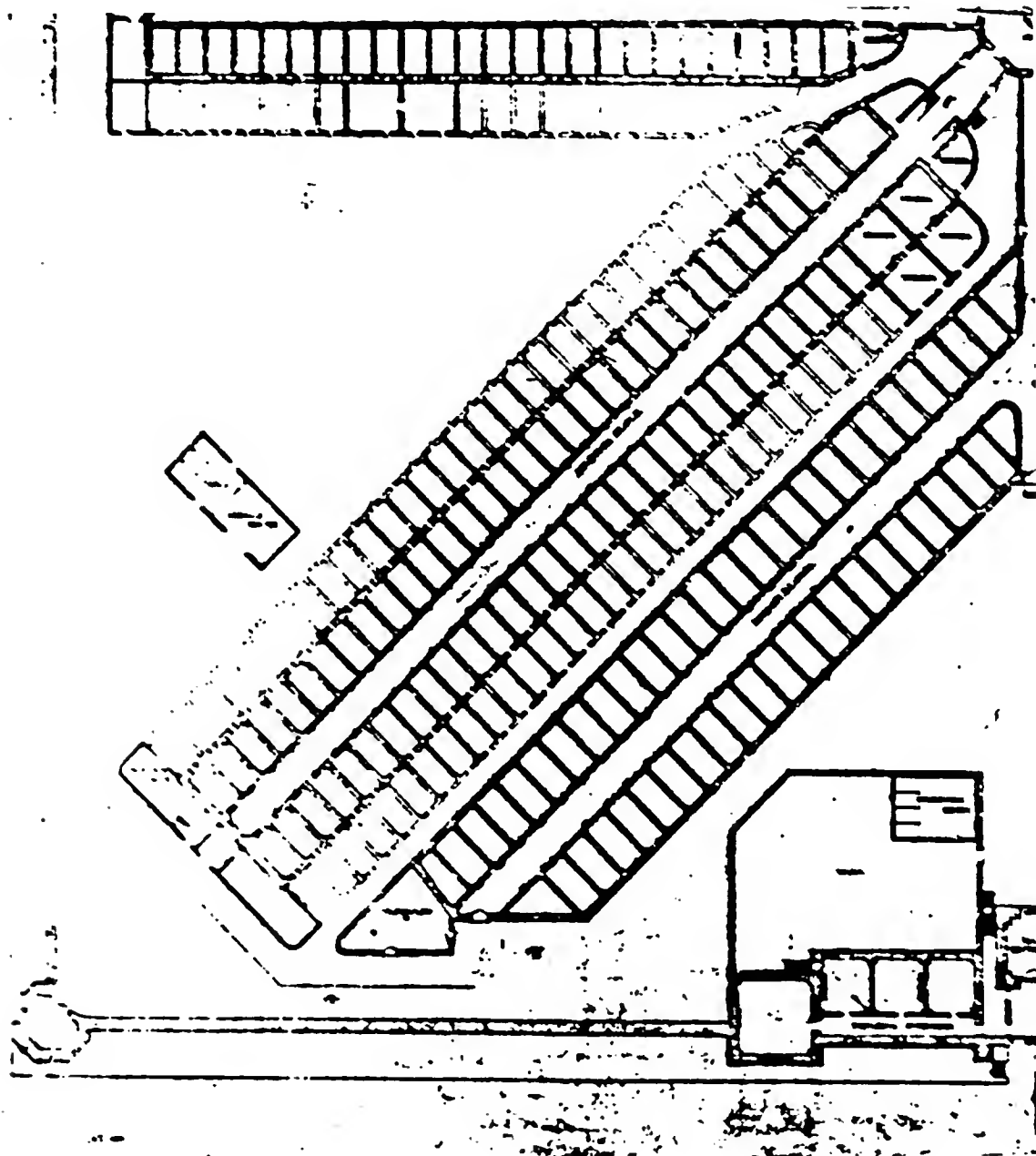
Marianna Thomas Architects, *Eastern State Penitentiary Historic Structures Report.* Philadelphia, 1994. Figure A16. George Spencer Morris and William S. Vaux, Jr., dels., "Survey of Present Buildings," 1 December 1900, Historical Society of Pennsylvania (Bc615 Ea77 [7]).

Figure 6: Second Floor Plan with Addition, 1900
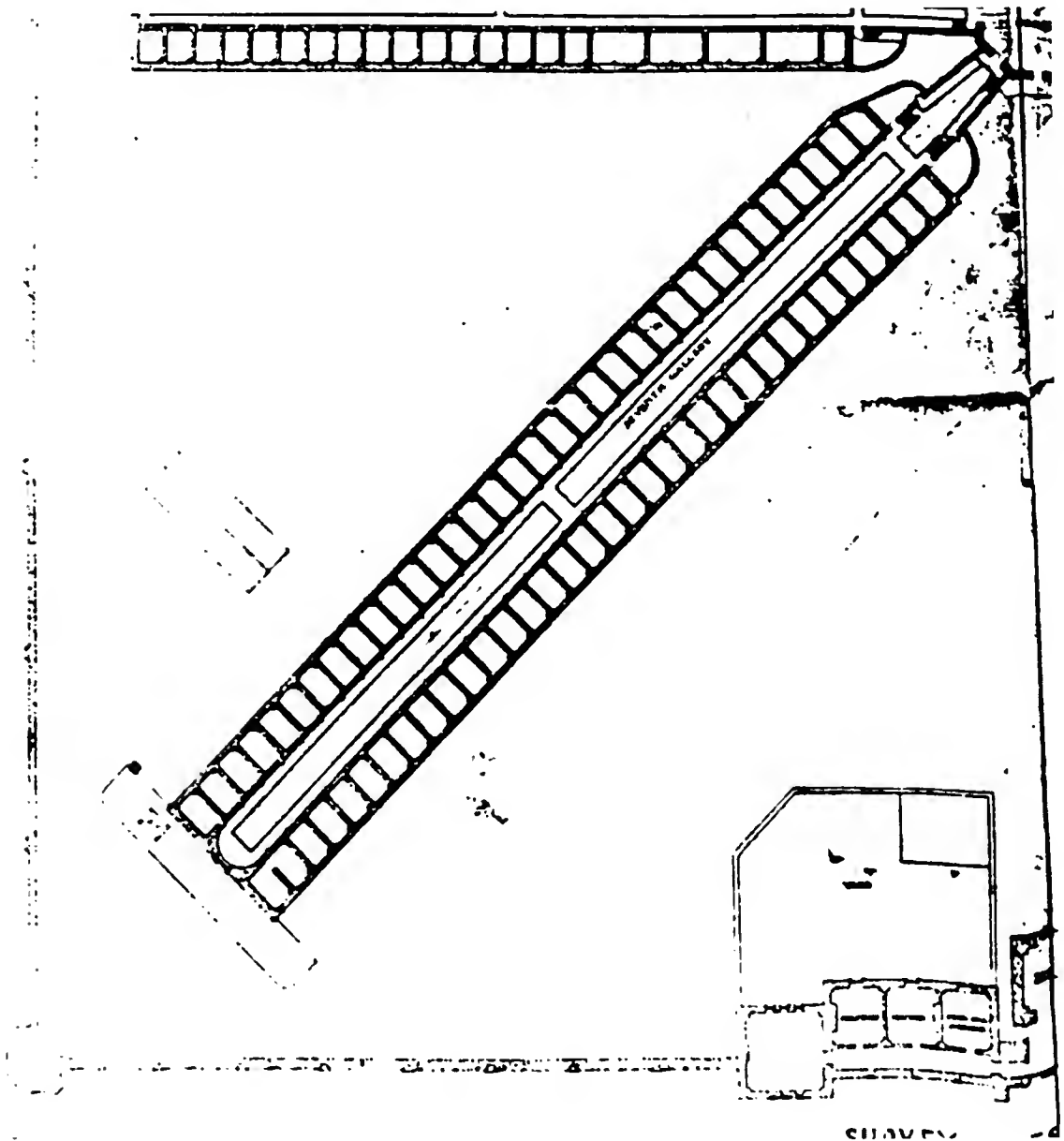
Marianna Thomas Architects, *Eastern State Penitentiary Historic Structures Report.* Philadelphia, 1994. Figure A17. George Spencer Morris and William S. Vaux, Jr., dels., "Survey of Present Buildings," upper storeys, 1 December 1900, Historical Society of Pennsylvania (Bc615 Ea77 [2]).

There is some question as to the impact of this addition on the exterior surface of the original structure. Of interest to this investigation is the possible application of gypsum plaster over the entire surface, which could be expected if the surface had at one time served as an interior wall. Such an application would contribute to the decay of the existing limewashed surfaces.

The nature of ancillary buildings within the penitentiary walls has been described as "minor timber-framed structures, including ... laundries and boiler rooms, attached to the principal buildings ... doubtless perceived of as temporary in nature [which] changed and disappeared accordingly."[16] This explanation suggests that the extant metal clamps visible on the facade may have served to attach such a timber-frame structure to the end wall, but that the impact would have been entirely mechanical, with no corresponding chemical effects as would result from the treatment of the end facade as an interior wall. This is supported by the observed conditions, which do not imply that any surface treatment has been applied to the upper level of the southwest facade.

A model of the penitentiary updated to circa 1904 no longer indicates the presence of the laundry addition, which had been moved in 1900 to another location within the complex.[17] No subsequent additions or alterations to the facade are reported.

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[16] Ibid., 91.
[17] Ibid., 189-190.

Figure 7: Model of Eastern State Penitentiary, 1904 Condition

Cell Block Seven is visible at the upper right, with the addition removed.
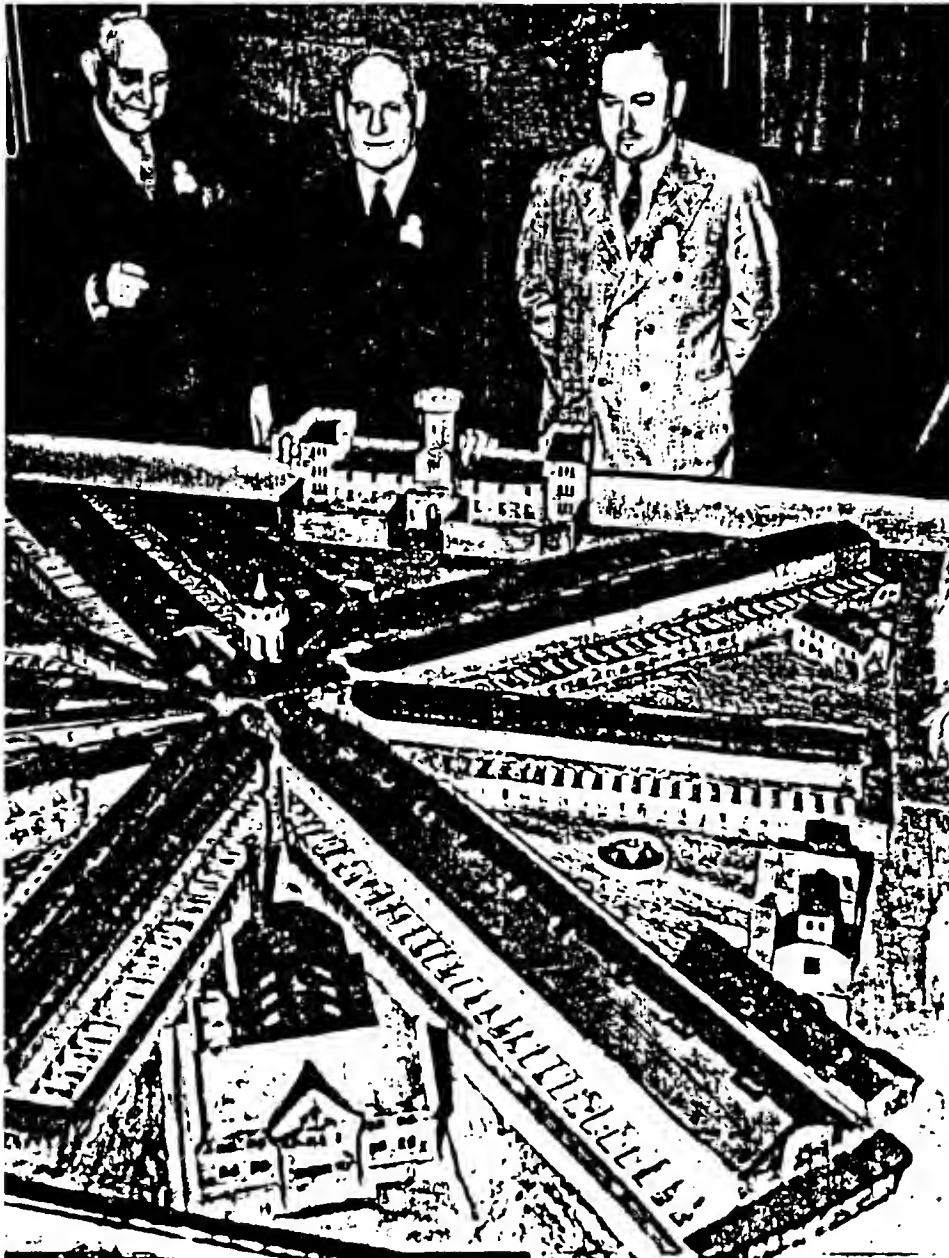
Marianna Thomas Architects, *Eastern State Penitentiary Historic Structures Report.* Philadelphia, 1994. Figure A20. 12 October 1937 photograph: *Philadelphia Record* archive, Historical Society of Pennsylvania.

Today, the southwest elevation is relatively intact, without residual additions or alterations to the original design. The upper level of the center wall appears remarkably sound, while the lower section and the east and west wing walls exhibit deterioration patterns that appear to be directly related to subsequent coating applications. This damage is particularly severe on the west wing wall, and there is speculation that this area was coated with a Portland cement earlier in this century. Only very small traces of limewash and patches that appear to be cement remain on the surface of this wall. The center and east wing walls both retain significant traces of limewash and other surface applications, including cement patches.

As this wall is the focus of the current study, existing conditions are discussed in greater detail in Section 4.1 of this report.

Chapter 3: Materials

3.1   Gneiss and Schist

Haviland's original design was executed in a variety of local materials. Among these are "best select stone" and "best Chester building stone",[18] which can be reasonably attributed to the large deposits of schist and gneiss in Delaware County. One account identifies the material of the Administration Building facade as granite, and the balance of the complex as executed in various gneisses and schists, including some extracted from the East Falls area, further identified as "Wissahickon schist, the archetypical Philadelphia foundation material until the early twentieth century..." [19], subsequently supplied by the Leiper quarry on Crum Creek, known as "Media stone"[20], of Delaware County.

3.1.1   Mineralogy

According to Stone's 1932 survey of Pennsylvania geology and quarry activity:

> Delaware County is abundantly supplied with building stone.... The igneous rocks are granite gneiss, hornblende gneiss, gabbro, and serpentine, and the sedimentary rocks are Baltimore gneiss and

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[18] John Haviland, perimeter wall cost estimate. Daybook no. 1. Haviland Papers, Van Pelt Library, University of Pennsylvania.

[19] Marianna Thomas Architects, *Historic Structures Report*, 89.

[20] Ibid.

Wissahickon gneiss. Large areas are underlain by gabbro, Wissahickon gneiss, and granite gneiss.[21]

Schist and gneiss, though identifiably different at the extremes of composition, comprise a continuum of manifestations of the same rock mineralogy. In general, gneiss is considered a superior building stone as it has finer grains and less mica, and is consequently more resistant to water infiltration and can take a smoother finish than can schist. The same minerals are found in the two rocks, in differing amounts and configurations. According to Eckel, "[t]he term gneiss is applied to rocks which have the same chemical and mineralogical composition as the granites and their allies, and which from their associations and occurrence are usually known to be of igneous origin; but in which the constituent minerals are arranged in roughly parallel layers or beds."[22]

Stone describes the materials as follows:

> Gneiss is a crystalline rock that has the same mineral composition as granite, and looks like granite, but the minerals have a more or less well-developed parallel arrangement, so that the dark minerals make noticeable black bands. There are several varieties of gneiss, named for the most prominent silicate. Gneiss differs from schist in the coarseness of the lamination, and by insensible gradations passes into schist.
>
> Schist is a rock composed mostly of quartz and mica arranged in parallel laminae or layers. If by variation the laminae become coarse and quartz predominates, the rock is called gneiss. Variation in the other direction, to

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[21] Ralph W. Stone, *Building Stones of Pennsylvania* (Harrisburg: Pennsylvania Geological Survey, 1932), 139.
[22] Edwin C. Eckel, *Building Stones and Clays: Their Origin, Characters and Examination* (Boston: Stanhope Press, 1912), 37.

very thin layers and mostly mica, becomes mica-schist. Schist tends to split in slabs, parallel to the laminae, and usually is rather soft, but as its component minerals are resistant to solution or alteration the rock is durable when protected from moisture and freezing.

Schist is a metamorphic rock produced by the alteration of either sedimentary or igneous rocks. ...It is named from the principal mineral, the commonest type in Pennsylvania being mica-schist...[which results] from the crushing and excessive shearing of granitic and other igneous rocks.[23]

For the purposes of this investigation, the material at Eastern State Penitentiary will be referred to as gneiss. It is generally finely grained with a granitic appearance. Some banding is visible, and laminar orientation is easily discerned. A great variety of quality and characteristics is evident in the material used at the site, but the area under investigation consists of relatively homogeneous material.

As gneiss is mineralogically similar to granite, this research draws heavily on published work that addresses the problems of granitic stones and their deterioration. One group in particular has published a great deal on this topic, with emphasis on the Leinster Granite of Ireland used in the Georgian buildings of Trinity College, Dublin.[24] These articles address questions of granite in conjunction with both Portland limestone and various

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[23] Stone, *Building Stones of Pennsylvania,* 3-4.
[24] See the publications of E. Bell, P. Boyland, T.P. Cooper, A.P. Duffy, S.J. Haneef, M.S. Jones, P.F. O'Brien, S. Pavia Santamaria, S.H. Perry, G.E. Thompson, and G.C. Wood for extensive discussions of the decay of Leinster granite in relationship to Portland limestone, mortars, and environmental factors.

mortars, and the contributions of these secondary materials to the deterioration of the granite. This collection of work has proven the most fruitful for the current investigation.

3.1.2 Weathering

3.1.2.1 Physical Weathering

Building stone is subject to deterioration from both physical and chemical processes. Physical deterioration usually manifests through fracturing at both a macroscopic and microscopic scale. In heterogeneous rocks, intergranular or interlaminar fractures can easily be seen with the naked eye, and the phenomena of flaking, spalling, sugaring indicate that this sort of large-scale physical damage is present. At the intragranular scale, individual grains of relatively non-reactive minerals can fracture as a result of stresses induced by thermal shifts, freeze/thaw, salt crystallization, and other material changes. When cracks develop at either scale, they provide a route for water infiltration and the further introduction of pollutants and salts.

3.1.2.2 Chemical Weathering

Granitic rocks are comprised of three major mineral groups: quartz, feldspars, and micas. As quartz is highly resistant to weathering, it is generally the other two groups that contribute most to the chemical deterioration of granitic rocks, including gneiss. This deterioration results from two major mechanisms: 1) the kaolinization of feldspars and 2)

water and pollutant infiltration at mica-rich sites as a result of that mineral's foliated

habit.   Biotites (dark micas with heavy iron content) seem to be particularly

problematic.[25]   Chemical deterioration is hastened by the presence of soluble salts, as

discussed by numerous authors.[26]

3.2   Lime and Limewash

Hydrated lime is a fundamental constituent of all plasters, mortars, and cements. It is an

strongly alkaline ingredient that contributes to the plasticity of the wet mixtures and to the

whiteness, softness and evaporative qualities of the cured materials. In its wet state, lime

has the composition $CaOH_2$, while after curing it returns to the chemical composition of

limestone, $CaCO_3$. The preparation and use of lime is well documented, and like stone

has been exploited for building since prehistoric times.

The question raised by the observed deterioration is clear: what characteristic or

characteristics of the limewash used here could explain such severe degradation? It has

been held traditionally that limewash alone provides a protective benefit to the masonry

to which it is applied. Limewash is generally considered to be a benign, sacrificial

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[25] See the publications of J.I. Drever and L.L. Stillings, R.M. Esbert et al., S.J. Haneef et al., F.M. Helmi, T.Y Irfan, M.S. Jones et al., J.G. Kelly, B.B. Lal, A. Perez-Ortiz et al., R.J. Schaffer, N. Schiavon et al., Petr Sulovsky et al., and S.A. Welch and W.J. Ullman for discussions of individual minerals and their dissolution or deterioration contributions.
[26] See Appendix A, Selected References: Salts and Masonry.

surface treatment with some consolidation properties -- self-healing due to recalcification

in cracks -- that protects the underlying material from direct water and pollutant

infiltration. Why, then, is the damage so aggressive in this case?

The following limewash recipes[27] may hold at least part of the answer to this question.

All of these formulas call for the addition of table salt to the mixture, which alone could

account for the observed damage. As described below, salt may have been added to assist

in the emulsification of other constituents and to retard evaporation, allowing for

complete carbonation in the relatively thin applied layer.

1. Whitewash used at the White House[28]: Receipt [*sic*] for Making White wash such as used on the Presidents house. Take half a bushel of unslacked [*sic*] lime, and slake it with boiling hot water, covering it over during the process. Strain it and **add a peck of salt dissolved in hot water**; three pounds of ground rice boiled to a thin paste put in boiling hot; half a pound of powdered Spanish whiting, and a pound of clean glue dissolved in water; Mix an let it stand for several days. Then keep in a kettle on a portable furnace and put it on as hot as possible with a painters or whitewash brush.

2. If the whitewash works poorly or comes out spotted, one ounce of potash alum dissolved in water and added to a pail of whitewash will correct the trouble, but too much drying whitewash that will not crack or peel can be made by slaking thirty pounds of builders' lime with hot water, keeping it steam for at least twelve hours. **When diluted add to the wash two pounds of zinc sulphate and one pound common salt** previously dissolved in water. It is not good practice to use glue as a binder for

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[27] All recipes have been kindly provided by Sally Elk, February, 1999.
[28] John Agg Papers c.1830: Special Collections Department, Duke University Library.

whitewash, and at any rate if the glue is not first class or has not been dissolved thoroughly, it will make the wash crawl and spot as you have stated. If you have to whitewash any surface, wall or ceiling that has been greasy it is best to wash it first with vinegar before applying the whitewash. It is an important point that brushes and pails, etc. are clean, and the wash should be strained.[29]

3. Limewashes: Slaked lime mixed with water will rub off rather easily and some additional ingredients are usually needed. Whiting (crushed chalk) and lime were traditionally mixed with glue, or size water, to bind them and improve adhesion. Sometimes, **common salt or crude commercial calcium chloride would be added** to tallow washes to assist the tallow to emulsify, and being hydroscopic would assist the carbonation of the lime on exposure. The common proportion was 15lb of common salt to 50lb of hydrated lime. These salt mixes are not recommended for historic fabric, quite apart from the risk of introducing unwelcome soluble salt crystallisation cycling, such mixes are not strongly adherent. Lime-glue formulations are another traditional type which should be discarded, even when improved with the addition of alum (for better working properties) and formaldehyde (for resistance to rubbing), as they require frequent maintenance.[30]

Moreover, feldspar dissolution rates change as a function of pH. Above pH 8, these rates increase with increasing pH.[31] In an alkaline microenvironment such as that produced by a limewash coat, this phenomenon is likely to affect granitic rocks of high feldspar content.

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[29] "Nineteen Ninety-five Paint Questions Answered," *Painters Magazine* (1919).
[30] John Ashurst, *Mortars, Plasters and Renders in Conservation*, 30.
[31] J.I. Drever and L.L. Stillings, "The Role of Organic Acids in Mineral Weathering," *Colloids and Surfaces A* 120 no. 1-3, (February 21, 1997): 167-181.

3.3   Portland Cement

Especially in the twentieth century, Portland cement has been used extensively for repointing and parging surfaces that had originally been served by lime mortars and stuccoes.   Such applications frequently result in severe degradation of the masonry material.   This degradation is due in part to the excessive hardness of Portland cement, which causes the stones to crack and spall under thermal stress; its low porosity, which results in the retention of water within the wall; and also the contribution of soluble salts from the cement to the stones. The sulfates contributed by Portland cement are addressed by the qualitative salt analysis performed in this investigation, although ettringite and other more exotic salts are not. The mere presence of cement on the wall is suggestive of additional deterioration mechanisms.   The contribution of Portland cement to the deterioration of Wissahickon gneiss and schist has been characterized as aggressive by Philadelphia conservators.[32]

3.4   Material Contributions to Deterioration

Salts containing chlorides and sulfates are known to induce severe deterioration in all types of building stone. Moreover, In granitic stones the presence of lime-rich mortars and stones has been proven to be detrimental. These elements provide soluble calcium

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[32] Author's conversations with John Carr and Sally Elk, November 1998 - March 1999.

which, in combination with sulfates from the atmosphere or other sources, forms gypsum crystals and crusts, which disrupt the surface and cause various decay forms.[33] [34]

The evidence provided by the above recipes, taken in conjunction with the physical manifestations of decay at the study site, suggests that common salt or other chlorides (for instance, calcium chloride) in the limewash may have contributed to the identified deterioration state. In addition, the presence of sulfates might be the result of added zinc sulfate. The most likely sources of sulfates, however, is gypsum formed by the reaction of soluble calcium from the mortars and limewash with airborne SO_2. Both chlorides and sulfates are present over the entire limewashed surfaces of the wall, as discussed in Chapter 5.

In addition to sodium chloride and gypsum, Portland cement used for patching and repointing has likely contributed to the damaged condition of the wall through increased mechanical stresses and the leaching of sulfates into the stone.

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[33] P.F. O'Brien et al., "Role of Mortars in the Decay of Granite," *Science of the Total Environment* 167, (May 1995): 103-110.

[34] T.P. Cooper et al., "Contribution of Calcium from Limestone and Mortar to the Decay of Granite Walling," In: *Science, Technology and European Cultural Heritage*, N.S. Baer, C. Sabbioni and A.I. Sors, eds., (Oxford: Butterworth, 1991), 456-461.

Chapter 4: Field Methodology and Results

4.1   Conditions Survey

In order to produce a record of the state of the subject elevation at the time of investigation, a conditions survey was executed.   The survey used decay type classifications developed by Quinlan, O'Daly and Lewis[35] to identify the visible conditions. See Appendix B for complete descriptions of these decay classifications.

Photographs of the facade were used to develop a template elevation in AutoCAD 14, with individual building stones detailed for the right center section.   Conditions were recorded on transparent overlays and drawn as layers over the template.

Figures 8 through 10 illustrate the conditions as of April, 1999.   Figure 8 shows the installed orientation of the individual stones, defined as either parallel or perpendicular to the laminar orientation of the material. As there is great variability in the orientation of individual blocks, a block was considered parallel if the angle between the laminae and a horizontal orientation was less than 45°. Any block displaying an angle greater than 45°,

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[35] Margaret Quinlan, Geraldine O'Daly and J. Owen Lewis, "Condition Assessment of Granite -- Exterior Facades," In: *Granite Weathering and Conservation: Proceedings of a Conference Held at Trinity College, Dublin, September 1993*, E. Bell and T.P. Cooper, eds. (Dublin: The Director of Buildings' Office, Trinity College, 1994), 46-56.

or for which the laminar orientation was not clearly discernible was considered to have a perpendicular orientation. Figure 9 shows the distribution of salt efflorescence on the surface of the wall. Figure 10 shows the distribution of the three physical deterioration manifestations observed on the wall: blistering, scaling, and relief.

The entire surveyed area shows moderate to severe damage. Traces of limewash are visible throughout, and at least one layer of surface treatment appears to match the remaining cement pointing mortar. In areas where the limewash is intact, the wall surface appears less prone to damage than in those where it has been breached. The limewash does not appear to have been applied over damaged stone, and it seems that the deterioration mechanisms it induces become more active and evident after a breach establishes a path for water penetration. The action of water in combination with other agents, such as soluble salts, leads to the dramatic deterioration observed in other areas.

Although the wall is severely damaged in some sections, and at least moderately damaged over the entire limewashed area, only the three degrees of deterioration illustrated in Figure 10 are present. The material is sufficiently damaged overall that areas of minor separation are quickly dislodged. Granular disintegration does occur, but only behind scales. This deterioration product is absent from the surfaces directly exposed to external weather conditions, and is therefore not recorded on the survey.

Figure 8: Block Orientation
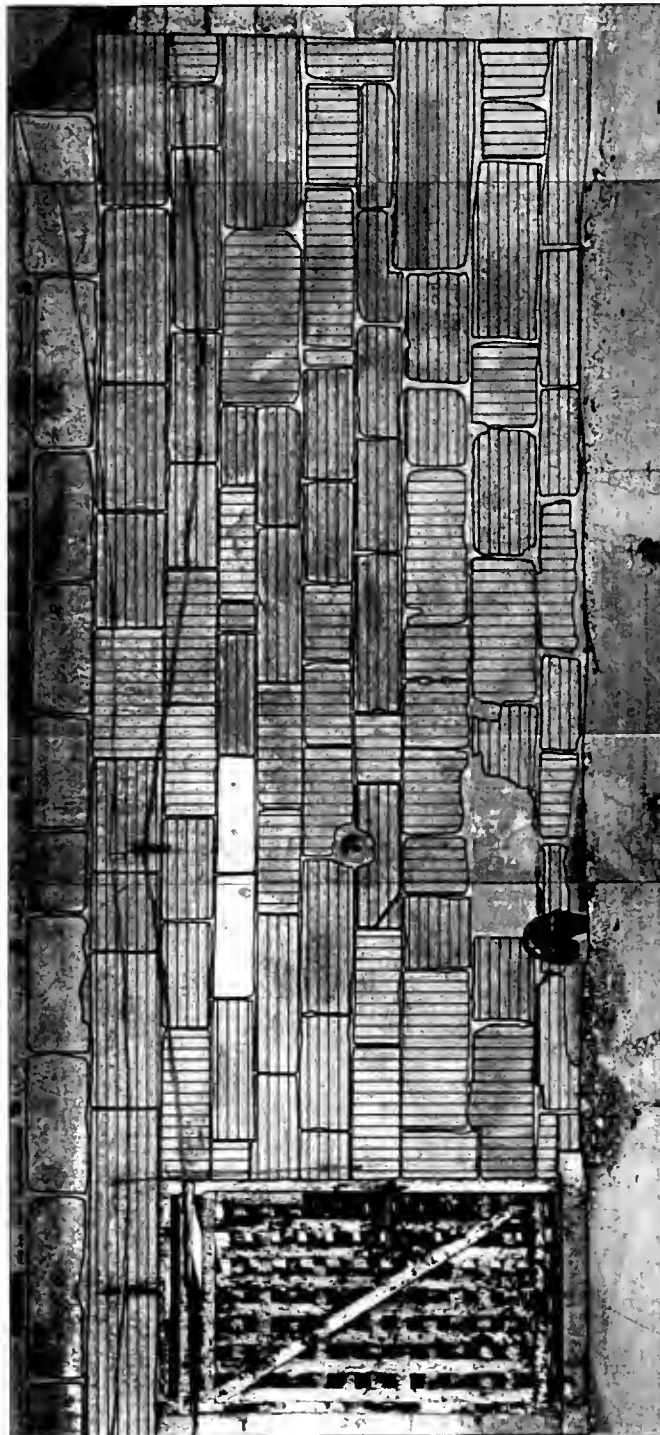
Figure 9: Salt Efflorescence
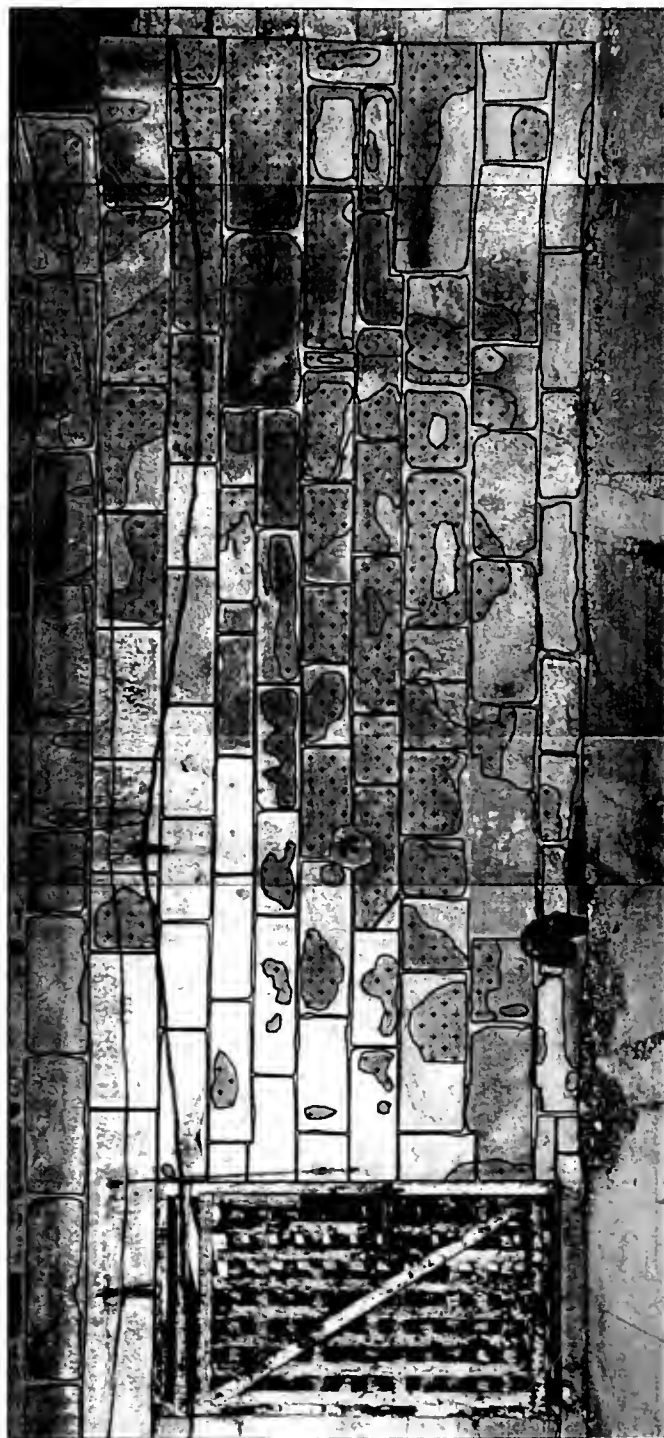
Efflorescence

Figure 10: Deterioration States
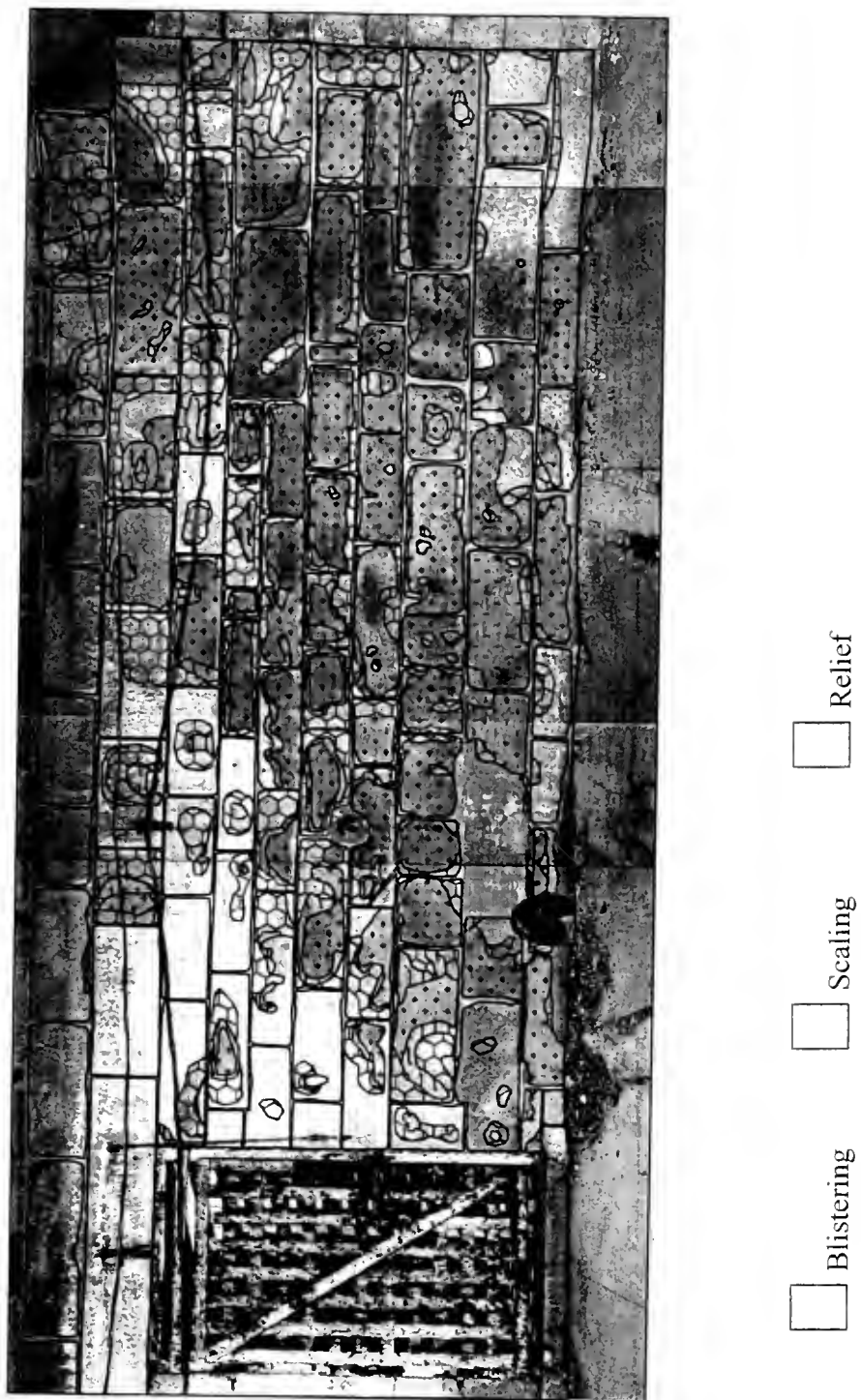
4.2   Chemical and Mineral Composition

In order to understand the mechanisms acting on the wall, it is necessary to understand the environment and the constituents being affected. The major components that require identification in this case are the mineral components of the stone and the anionic constituents of the soluble salts present.

Mineralogy was investigated by the use of thin section microscopy, X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). Salt identification was investigated by SEM/EDS and wet chemical analysis of the soluble portion.

4.2.1   Thin Sections

For this investigation, thin sections were prepared from six samples: two deterioration crusts without limewash, two deterioration crusts with limewash, and two samples of sound stone. Thin sections were cut perpendicular to the weathered surface in order to illustrate the differences from exterior to interior surfaces.

Table 4.1: Thin Section Sample Sources

Sample No.	Type	Limewash	Date Taken	Location
1	deteriorated crust	no	12/98	northwest wing wall
2	deteriorated crust	no	1/99	
3	deteriorated crust	yes	1/99	southeast wing wall
4	deteriorated crust	yes	1/99	ground
5	solid fragment	no	12/98	perimeter wall, ground
6	solid fragment	some	1/99	trash rock

This analysis revealed that the stone in service at Eastern State Penitentiary, although considered granitic, has an low quartz content. The major constituents of the stone are feldspars, with a heavy microcline content, and micas, including biotite and muscovite. Both microcline and biotite are particularly vulnerable to deterioration. Figures 11 through 16 illustrate the samples under crossed nicols at 25x magnification. In all photomicrographs in this report, the outer edge of the scale bar is equivalent to 1.4 mm.

Figure 11: Sample 1

Deteriorated Crust, No Limewash
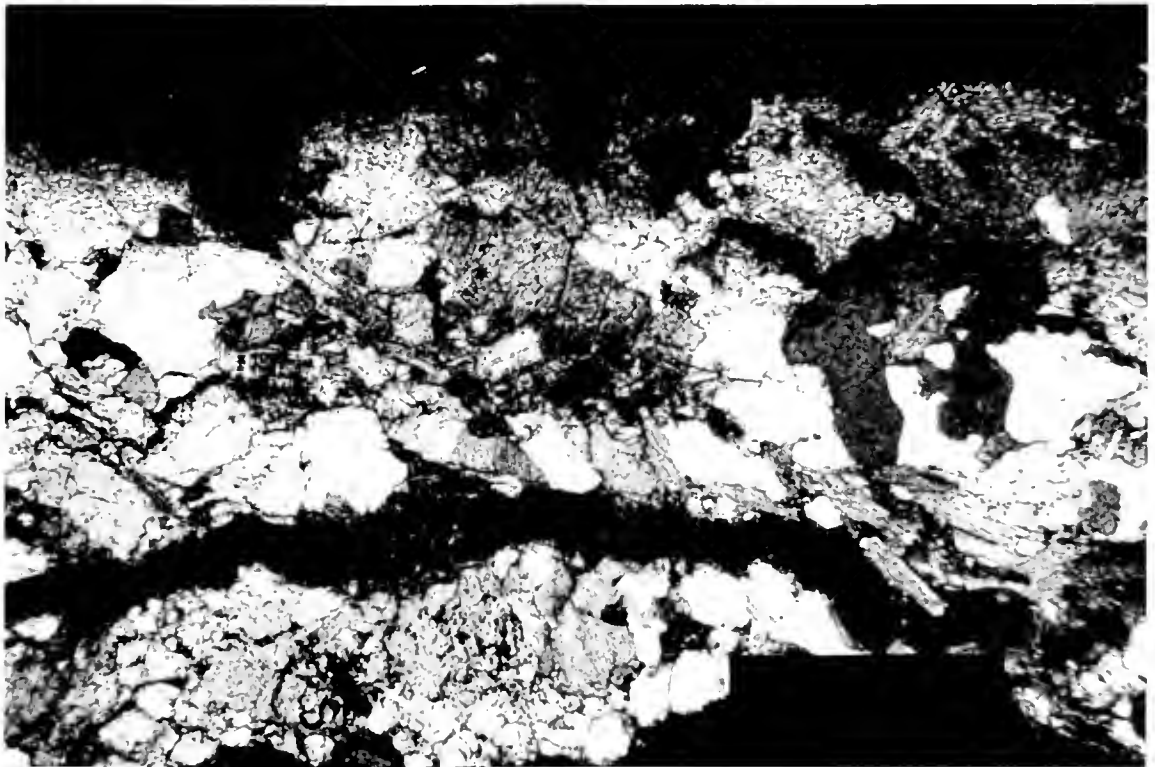
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 12: Sample 2

Deteriorated Crust, No Limewash
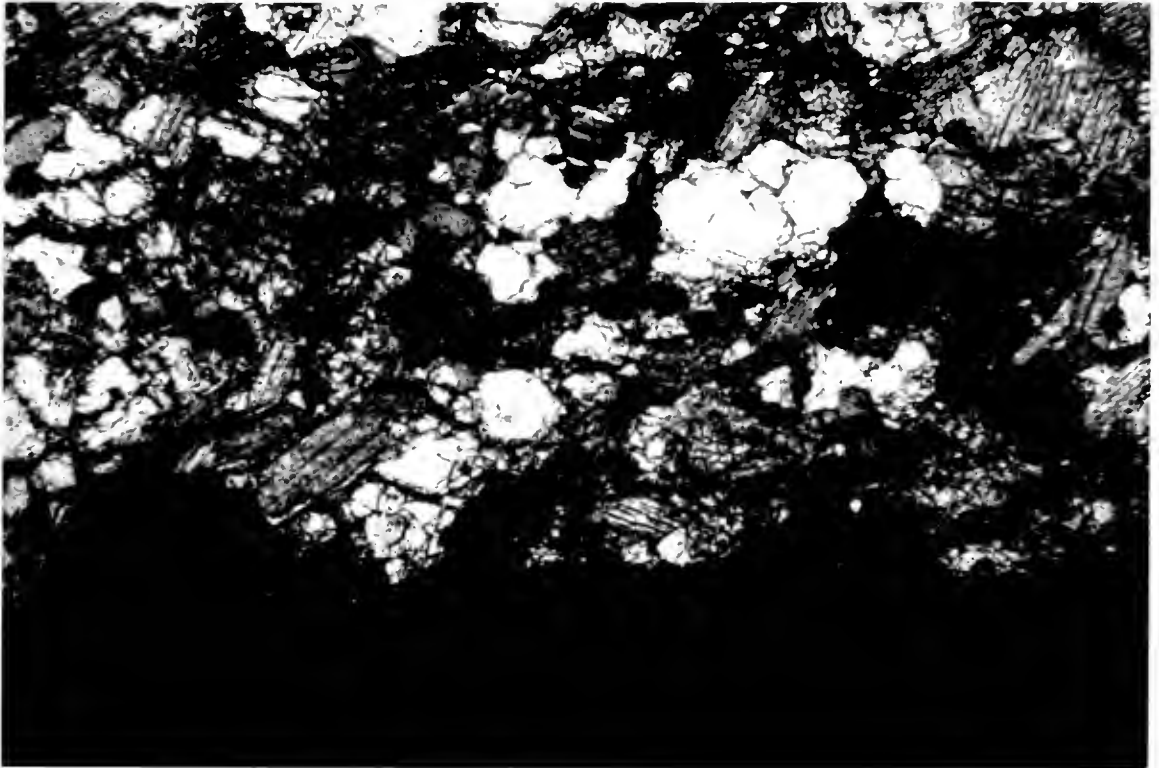
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 13: Sample 3

Deteriorated Crust, With Limewash
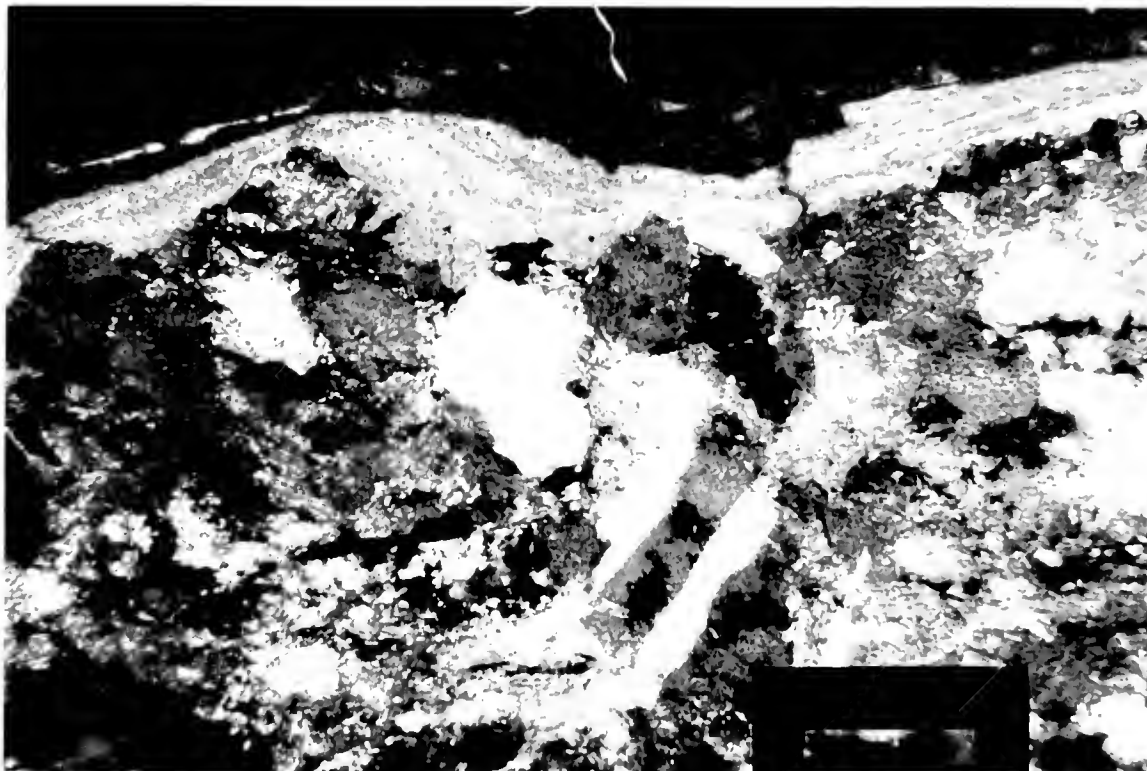
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 14: Sample 4

Deteriorated Crust, With Limewash
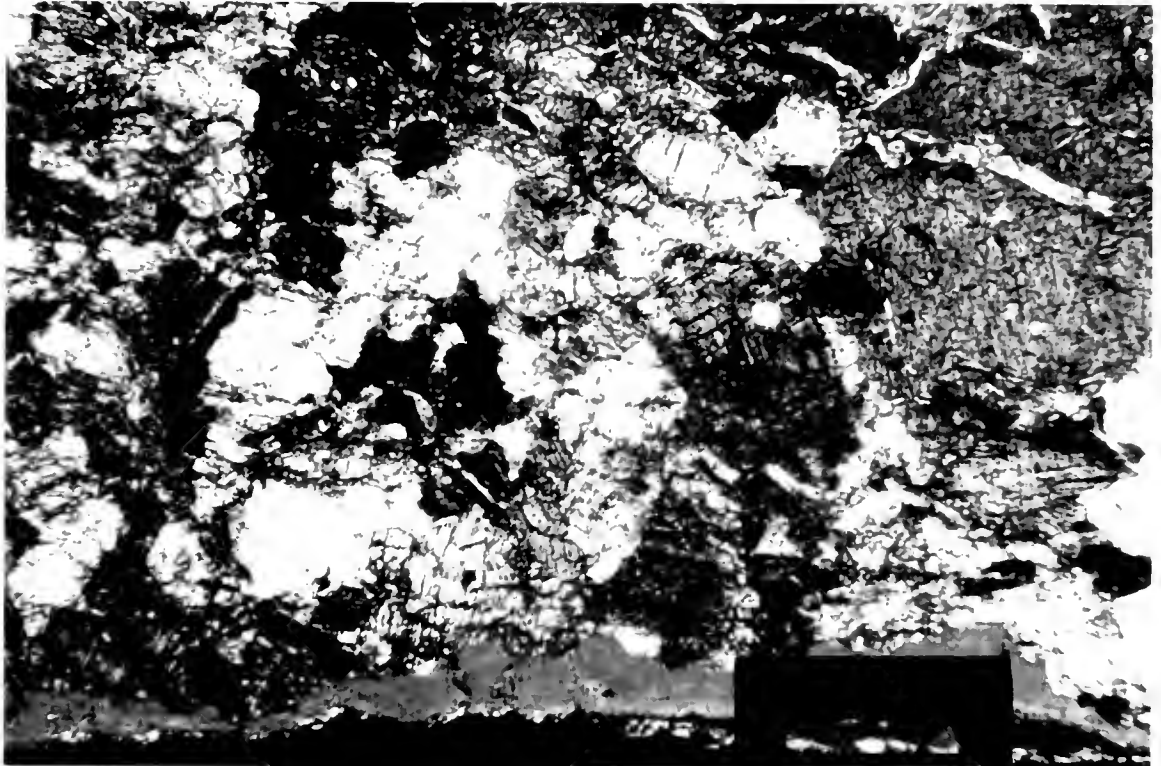
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 15: Sample 5

Solid Fragment, No Limewash
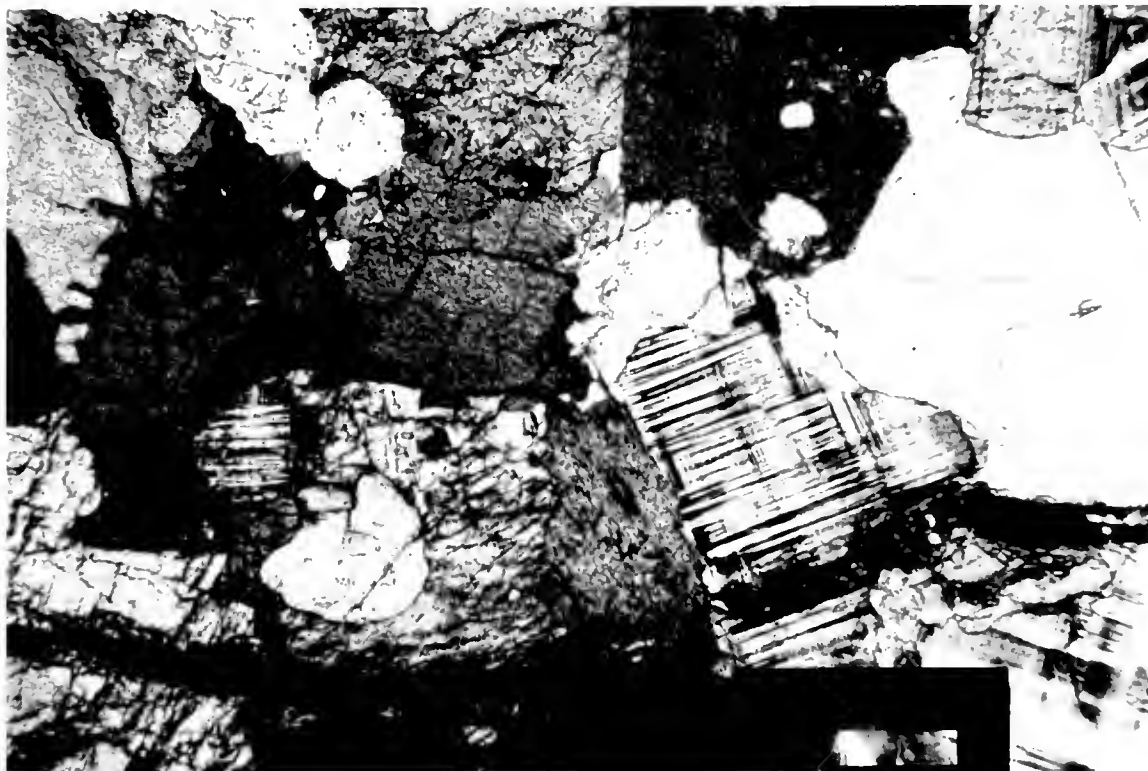
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 16: Sample 6

Solid Fragment, Some Limewash
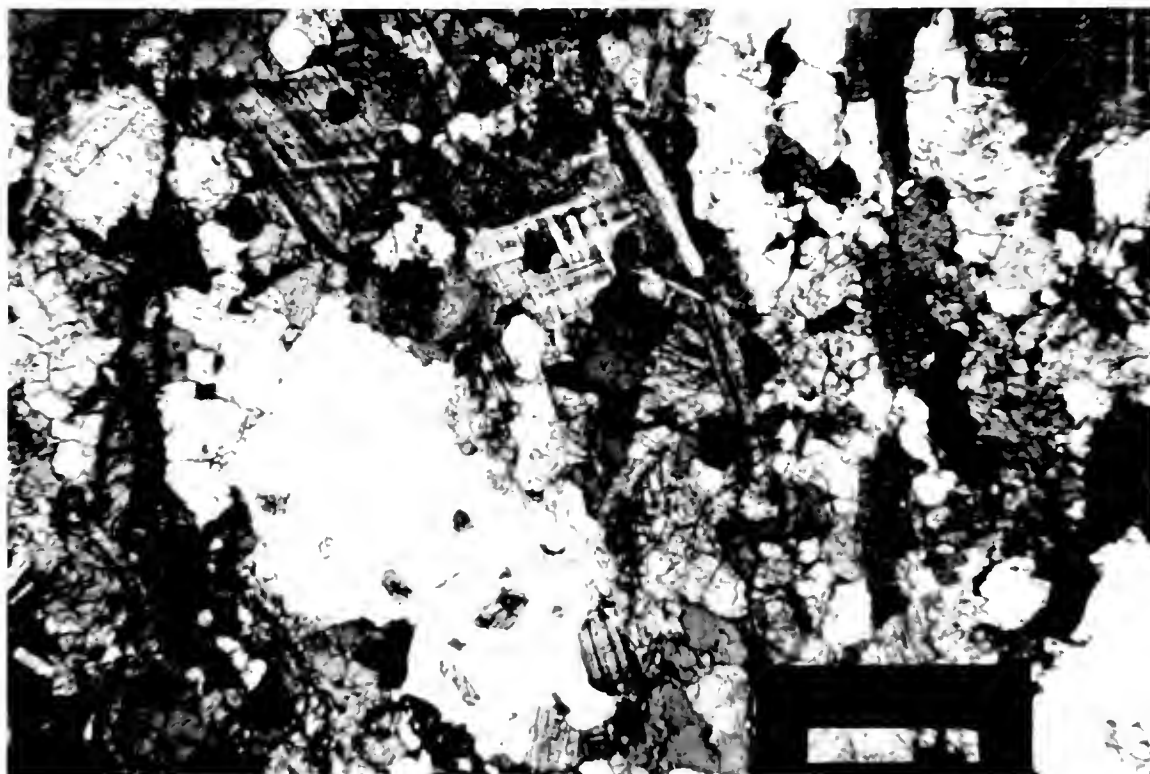
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

4.2.2   XRD

X-Ray Diffraction (XRD) is used to identify the crystalline structure of constituent materials present in a given sample. XRD was carried out on one sample of deterioration crust to verify mineralogical composition. Figure 17 shows the results of this test, which was conducted on Sample 2.

The crystalline materials identified include quartz and biotite, with a possible identification of sanidine.

Figure 17: XRD, Sample 2
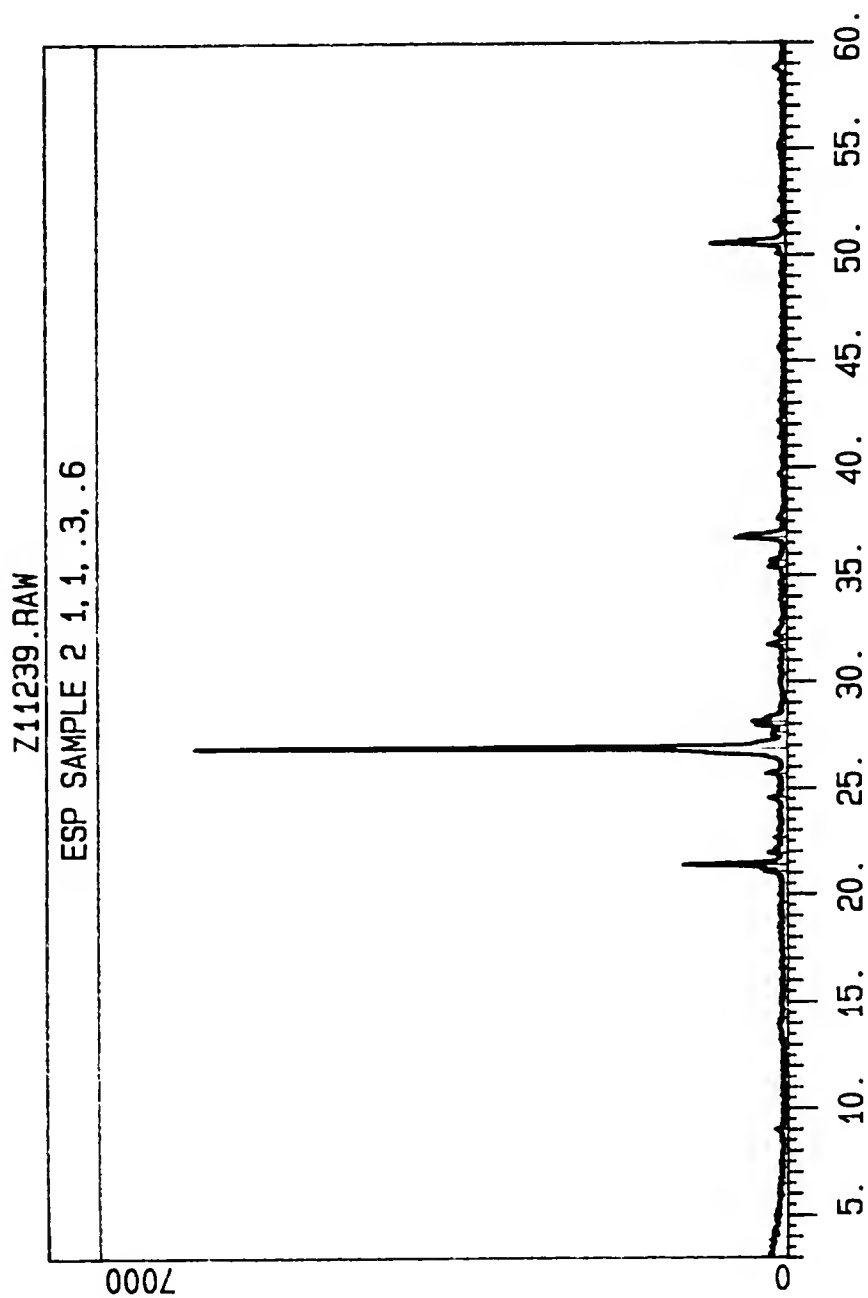
4.2.3   SEM/EDS

Scanning electron microscopy (SEM) was used to visually verify the presence of constituent minerals (quartz, feldspar, mica) in the base stone, and Electron Dispersive Spectra (EDS) to identify elements in deterioration crust and limewash.   The identification of chlorides would support the theory that salt was added to the limewash used in this area.

Figure 18 is an image of Sample 3 taken under SEM, showing the three constituent minerals of gneiss: feldspar, mica and quartz.

Figure 19 includes the overlaid EDS results from two different limewash surfaces on Sample 3. Although chlorine does appear, it is not a major constituent of these samples. This finding does not invalidate the theory that sodium chloride was added to the limewash, however, as this salt is extremely soluble and could easily have leached out of the material upon coming in contact with liquid water.

Figure 20 shows the results of EDS run on mica of the deterioration crust in Sample 2 without limewash present. The high readings for aluminum and silica are consistent with the presence of mica, and the presence of potassium indicates this particular mica is muscovite.

Figure 18: SEM, Sample 3

This is a Polaroid image of Sample 3 taken under SEM clearly showing the constituent minerals: mica, feldspar, and quartz. The mica is recognizable by strong foliation, the feldspar by characteristic cleavage lines, and the quartz by conchoidal fracture.
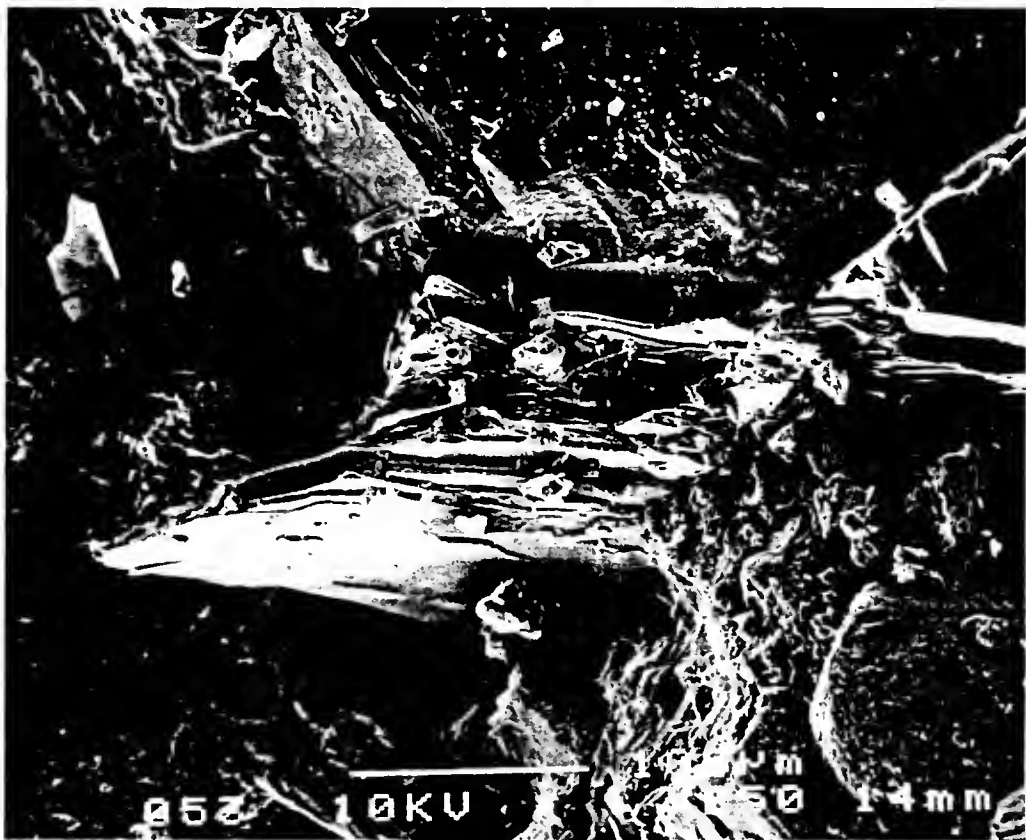
Photo: R. Lakis

Figure 19: EDS, Sample 3 Limewashes
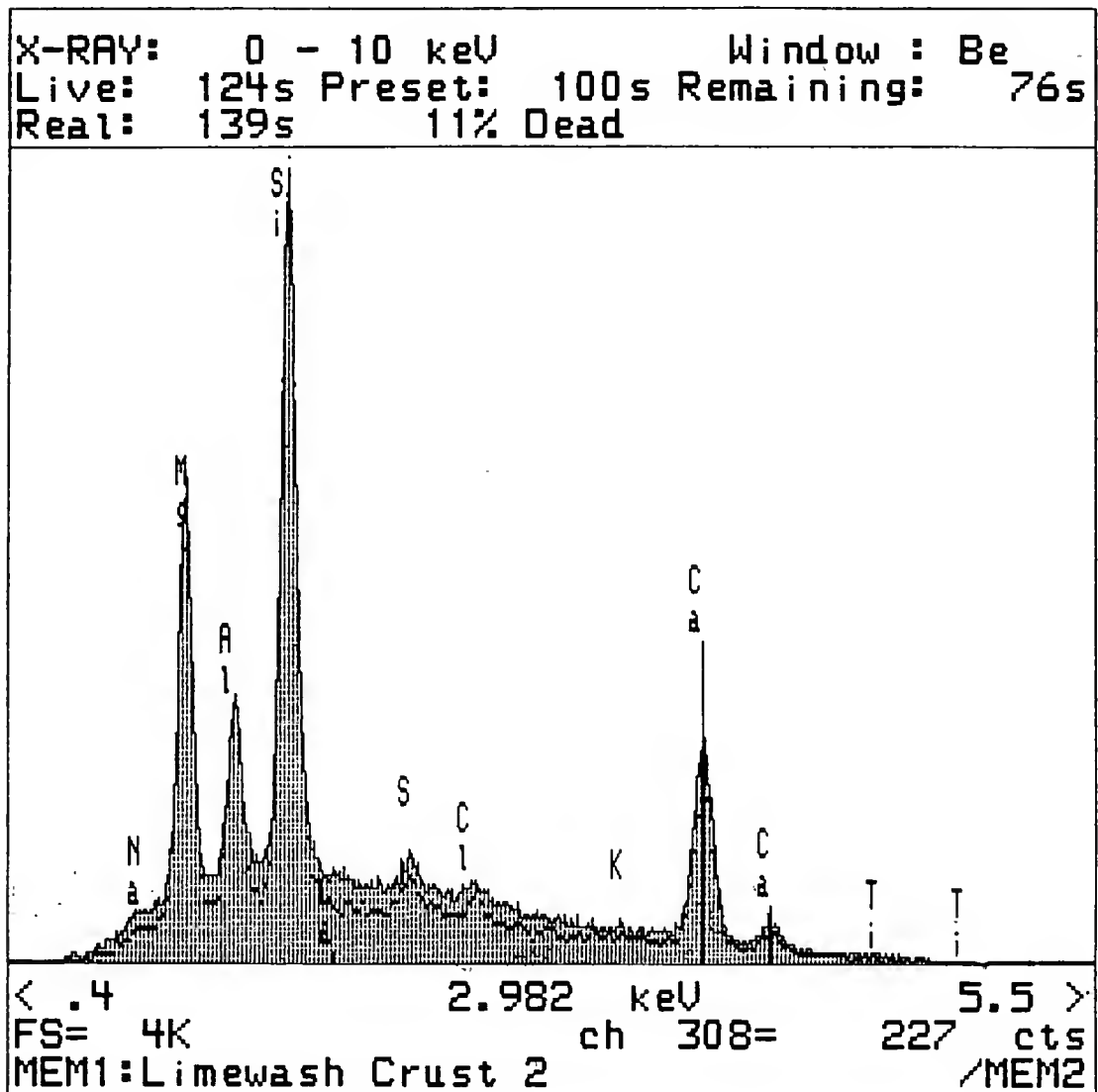
Figure 20: EDS, Sample 2 Deterioration Crust - Mica
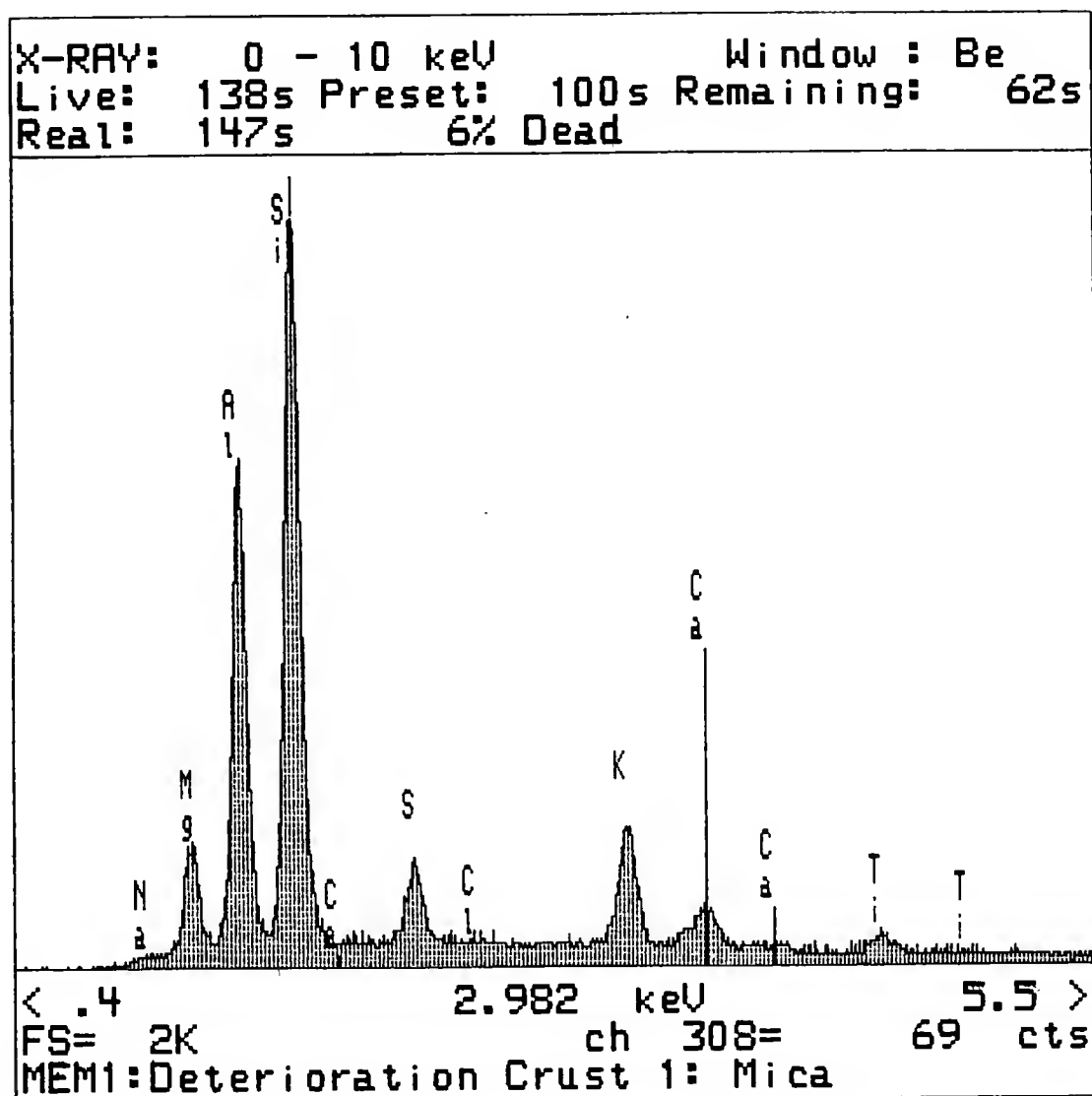
4.2.5 Qualitative Salt Analysis

Qualitative salt analysis was carried out on thirty samples from the Cell Block Seven elevation. Two samples, one with and one without limewash, were taken from three elevations at five points across the three wall elevations. One set was taken from each of the wing walls (six samples per wall), and three sets at the east, center, and west, were taken from the center wall.

All samples were analyzed according to the procedure published by ICCROM in 1988.[36] Results were recorded for the presence of sulfates, chlorides, nitrites, nitrates, and carbonates.

All of the soluble salts were present in at least one sample. Nitrates and nitrites were found only occasionally, and only just at or near the limit of perception. Predictably, carbonates were found most strongly in the limewashed samples. Chlorides and sulfates were present at all elevations and across the tested area.

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[36] Jeanne Marie Teutonico, *A Laboratory Manual for Architectural Conservators* (Rome: ICCROM, 1988), 113.

Table 4.2: Qualitative Analysis of Salts

Sample	Location*	Limewash	Sulfates	Chlorides	Nitrites	Nitrates	Carbonates
C1	C, W, L	No	+	+	-	-	+
C2	C, W, L	Yes	++	+/-	-	-	+++
C3	C,W,M	No	+/-	+	-	-	+/-
C4	C,W,M	Yes	+	+/-	-	-	+++
C5	C,W,H	No	+	+/-	-	-	+
C6	C,W,H	Yes	+	+/-	-	-	++
C7	C,M,L	No	+/-	+/-	+/-	-	-
C8	C,M,L	Yes	+/-	-	-	-	++
C9	C,M,M	No	+	+	+/-	-	-
C10	C,M,M	Yes	+	+	-	-	+++
C11	C,M,H	No	-	+	-	-	+/-
C12	C,M,H	Yes	-	+/-	-	-	+++
C13	C,E,L	No	-	-	-	-	-
C14	C,E,L	Yes	++	+	+	-	+++
C15	C,E,M	No	+/-	-	-	-	+
C16	C,E,M	Yes	+/-	+	+/-	-	+++
C17	C,E,H	No	+/-	+/-	-	-	+/-
C18	C,E,H	Yes	+	+	-	-	+++
W1	W,L	No	++	+	-	-	+
W2	W,L	Yes	++	+	-	-	+++
W3	W,M	No	-	+/-	-	-	+
W4	W,M	Yes	+/-	+	-	-	+++
W5	W,H	No	+/-	+	-	-	++
W6	W,H	Yes	+/-	+/-	-	-	+++
E1	E,L	No	++	+	-	-	+
E2	E,L	Yes	++	+/-	-	+/-	+++
E3	E,M	No	+++	+	-	-	+
E4	E,M	Yes	++	+/-	+	-	+++
E5a	E,H	No	++	+	-	-	+
E6b	E,H	Yes	++	+/-	+/-	-	+++

C: Center Wall   L: Low Elevation (>1 meter above grade)
W : West Wing Wall   M: Middle Elevation (1-2 meters above grade)
E: East Wing Wall   H: High Elevation (2+ meters above grade)

The presence of chlorides indicates the strong likelihood that sodium chloride was used in the whitewash. Were deicing salts the sole source of chlorides, they would only be found up to a height of approximately one meter. As shown above, the ion is found up to a height of two or more meters, which is inconsistent with salt transport by capillary rise. Thus we may conclude that sodium chloride was used in the limewash applied to this wall. Similarly, the presence of sulfates indicates the likelihood that Portland cement was applied to this surface at some point in the past. The salt test is corroborated by the visual inspection, which revealed traces of cement in an earlier limewash layer.

Chapter 5: Laboratory Methodology and Results

5.1   Sampling and Sample Preparation

Two types of samples were taken from the site for the analytical program. For this study, the term *hand sample* refers to material taken directly from the wall and its immediate vicinity in order to determine the existing conditions in situ. Deteriorated hand samples were taken from the end wall of Cell Block Seven for analysis in thin section and for qualitative salt testing. These samples have been discussed in Chapter 4.

The term *bulk sample* refers to cut, cleaned stone cubes that were subjected to laboratory weathering and analytical tests in an attempt to determine some of the behaviour characteristics of the material under various controlled conditions. Blocks of sound stone were obtained from a pile of discarded building stone north of Cell Block Five. From this sound stone, bulk samples of approximately 5cm x 5cm x 5cm were cut using a Frantom Rock Saw (oil cut) and a Raytech Table Saw (water cut). After cutting, the blocks were laid in clay cat litter to absorb surface oil, scrubbed with Dawn Extra detergent, and soaked in a fresh detergent solution for twelve hours. The samples were then rinsed under running warm tap water for twelve hours. A second soak period of six hours was followed by second twelve-hour rinse.

The washed samples were dried at 100°C for 48 hours, and cooled in a dessicator conditioned with Drierite. Of the sixteen samples prepared, fifteen were used in various tests and one remained untested. Table 5.1 summarizes the samples and their treatments.

Table 5.1: Samples and Treatments

Sample	Lime Soak	Gypsum Soak	Salt-Gypsum Soak	Gypsum Evap.	Capillary Rise	Total Immersion	Drying Index	Thin Section
A		x				x	x	
B				x			x	
C					x		x	
D		x				x	x	
E					x		x	
F			x				x	
G	x							x
H	x						x	
I				x			x	
J			x				x	
K					x		x	
L				x				x
M		x						x
N			x					x
O	x						x	
P					x		x	
Q								x

5.2   Physical Properties

5.2.1   Bulk Density

Bulk density is the ratio of mass to total volume.   As the samples are not perfect cubes,

all samples were measured with calipers to determine the length of every edge.   The

volume of each sample was calculated as the cube of the mean of all side lengths.   The

mass of each sample was measured after cooling in a Drierite-conditioned environment.

Table 5.2: Bulk Density

Sample	Mean Depth (cm)	Mean Width (cm)	Mean Height (cm)	Volume (cm^3)	Mass (g)	Bulk Density (g/cm^3)
A	5.114	4.453	5.085	115.780	300.30	2.59
B	5.130	4.451	5.164	117.914	307.06	2.60
C	5.246	4.788	5.439	136.602	363.00	2.66
D	4.976	4.889	4.979	121.121	313.17	2.59
E	5.517	4.671	4.671	137.174	361.82	2.64
F	4.676	4.911	4.473	102.716	264.45	2.57
G	4.794	4.176	4.785	95.795	252.73	2.64
H	4.999	4.614	5.030	116.007	299.53	2.58
I	4.894	4.360	4.835	103.163	264.89	2.57
J	4.675	5.088	4.938	117.434	304.38	2.59
K	4.936	5.214	4.953	127.459	327.43	2.57
L	4.481	4.920	4.641	102.329	259.90	2.54
M	4.459	4.859	4.286	92.857	235.87	2.54
N	4.841	4.429	4.854	104.068	269.30	2.59
O	4.624	5.340	4.800	118.516	306.34	2.58
P	4.361	4.208	4.513	82.804	214.36	2.59
Q	4.828	3.593	4.730	82.031	206.76	2.52
Mean	*4.856*	*4.645*	*4.834*	*110.222*	*285.37*	*2.59*

5.2.2   Capillary Water Absorption

This characteristic provides an indication of the behaviour of the material under investigation when it is in direct contact with liquid water at one surface.   A laminar material, such as heavily foliated gneiss, might exhibit differential behaviour as a result of orientation to foliation planes.   This test may suggest how the material would respond to a heavy rain, water infiltration from the soil, or some other constant water source at the exposed surface.   This experiment was run following NORMAL 11/85 (see Appendix C.1).

The results from Samples E, P and K are presented below.   Sample C produced anomalous results and was disregarded as a control sample. See Appendix D.5 for more information on Sample C.

Although both Sample E and Sample P were tested with the foliation plane in the direction of capillary rise, the data from this test do not indicate a significant difference in results as a function of foliation plane orientation. The results for Sample K, which was tested with the foliation plane against the direction of capillary rise, fall within the range set by E and P.

Capillary Water Absorption
Samples E and P: With Foliation
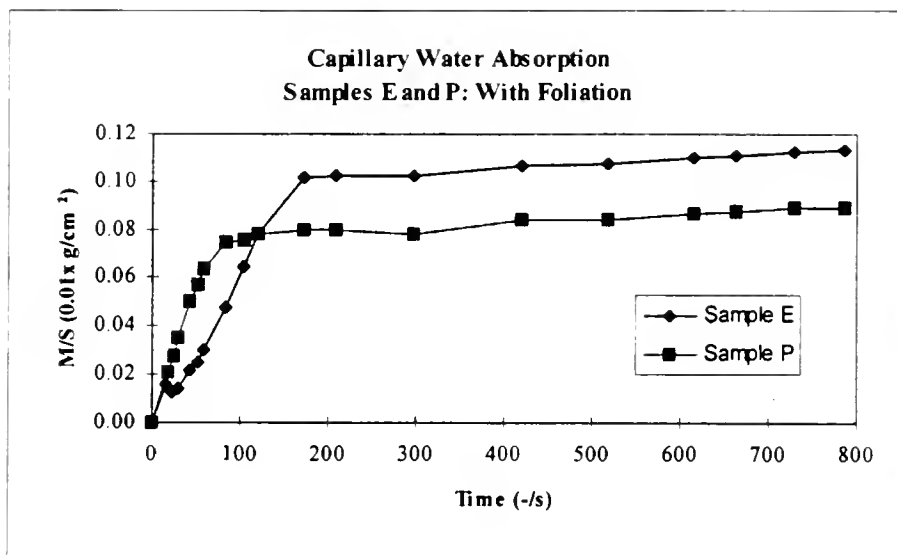
When the first reading was taken at t=4 minutes, Sample E was not blotted to remove surface water. This experimental error accounts for the step in the curve.

Capillary Water Absorption
Sample K: Against Foliation
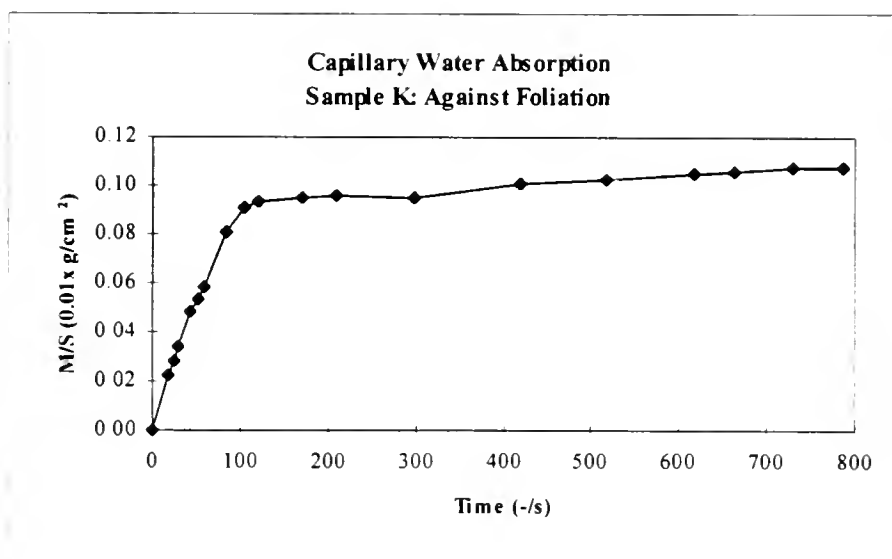
5.2.3 Water Absorption by Total Immersion

This test indicates the behaviour of the material under investigation when it is in contact with liquid water on all sides.   For the purposes of this investigation, samples were immersed after the test for Water Absorption by Capillary Rise was completed, in order to assure complete saturation for the Drying Index.

5.3   Experimental Weathering

5.3.1   Immersion

Three samples each were immersed in saturated solutions of lime ($Ca(OH)_2$), gypsum ($CaSO_4.2H_2O$), and table salt ($NaCl$) and gypsum, for a minimum of 12 hours per day for the four week test period.   During cycling, the samples were removed from immersion and dried in a closed chamber with Drierite for between five and twelve hours, then returned to the solution.   The pH of each soaking solution was measured daily, and the solutions were changed weekly.

5.3.2   Evaporation

Three samples were placed in sealed containers so that the samples' bases were immersed in a saturated gypsum solution while the upper surface was open to the air.   The samples were left in this setup for one month in order to develop a gypsum crust at the upper surface through evaporation of the solution through the samples.

5.4   Testing and Evaluation of Weathered Samples

5.4.1   Drying Rate

This test, based on NORMAL 29/88 (see Appendix C.2) measures the rate at which the water-saturated material loses moisture through evaporation. The results of this test, plotted as a curve, typically show a steep weight loss early in the testing period that is followed by a shallower curve after the early moisture is lost. Bulk samples of all treatments were tested to determine the changes in drying behaviour introduced by the various solutions.

The following charts show the results of this test. The value Qi in the graphs represents the residual water content at the time of measurement as a percentage of the original dry weight of the sample.

Both the lime-soaked and the salt/gypsum-soaked samples produced drying curves significantly different from those of the untreated samples. This may be explained for the former by the microfractures induced by the weathering process (see section 5.4.3 for further explanation of this and other weathering-induced phenomena). The increased microporosity could account for the increased drying time. In addition, the thin layer of carbonated lime that developed on the exposed surfaces may have produced some blocking action. The behaviour of the salt/gypsum-soaked samples may be explained by

the increased total porosity induced by weathering and by the hygroscopicity of the salt itself.

The gypsum evaporation samples produced drying curves somewhat different from those of the untreated samples, and the gypsum-soaked samples behaved similarly to the untreated samples. The blocking action of the gypsum crusts on the gypsum evaporation samples may account for the change in their behaviour[37], while the low solubility of gypsum may account for the lack of change in the gypsum-soaked samples. In the case of the gypsum evaporation samples, one full month was allowed for the formation of the crusts. Soaked samples experienced only short daily drying cycles, which did not favor the formation of thick surface crusts.

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[37] L. Franke and J. Grabau, "Influence of Salt Content on the Drying Behaviour of Brick," In: *Conservation of Historic Brick Structures*, N.S. Baer, S. Fitz and R.A. Livingston, Eds. (Donhead: Shaftesbury, 1998), 59-68.

Drying Curves: Untreated

Drying Curves: Lime Soak

Drying Curves: Gypsum Soak

Drying Curves: Salt/Gypsum Soak

Drying Curves: Gypsum Evaporation
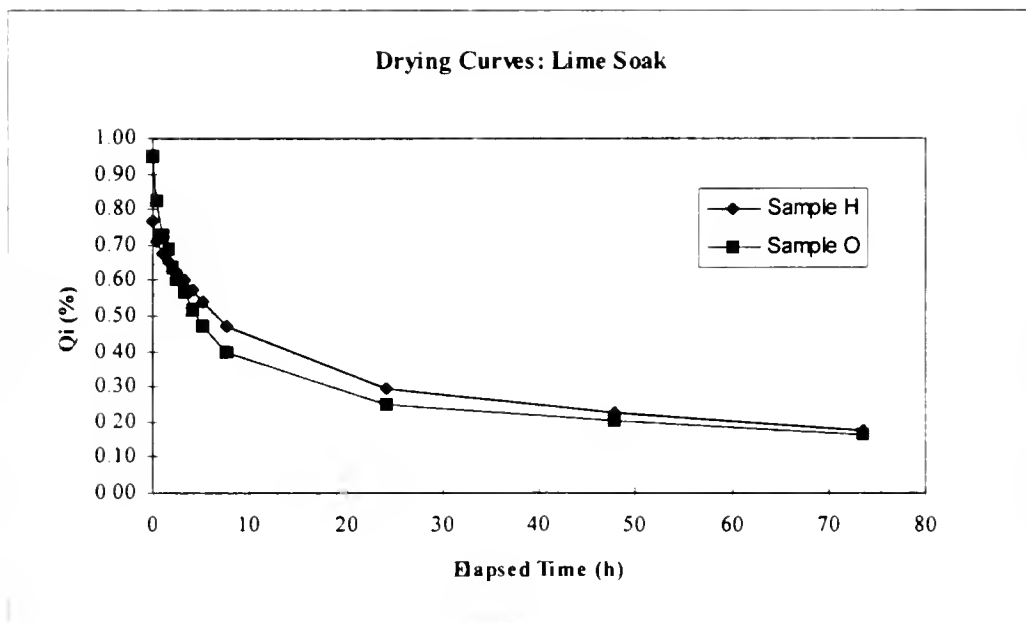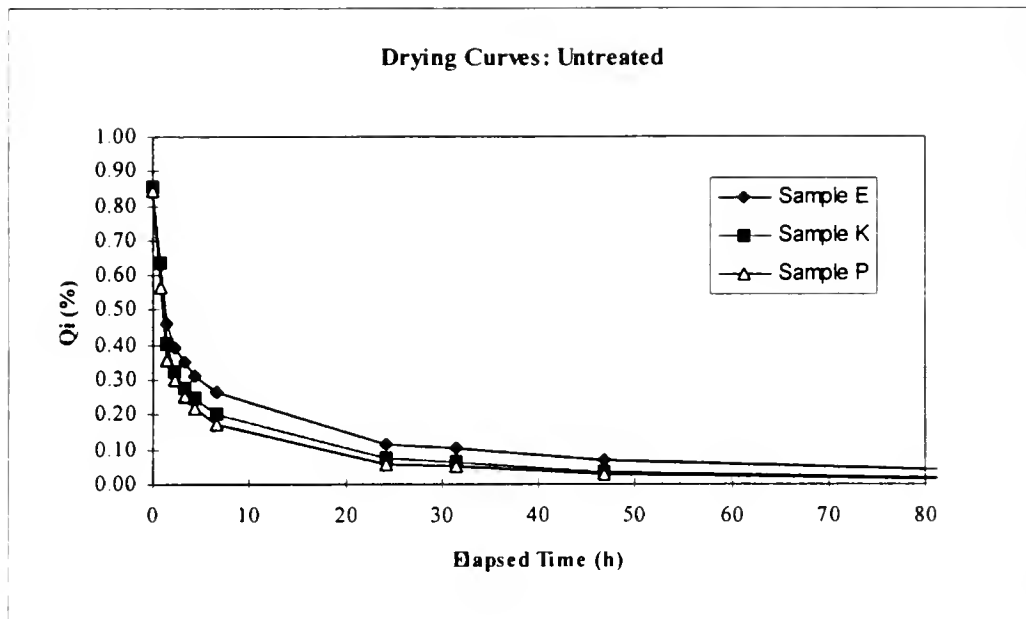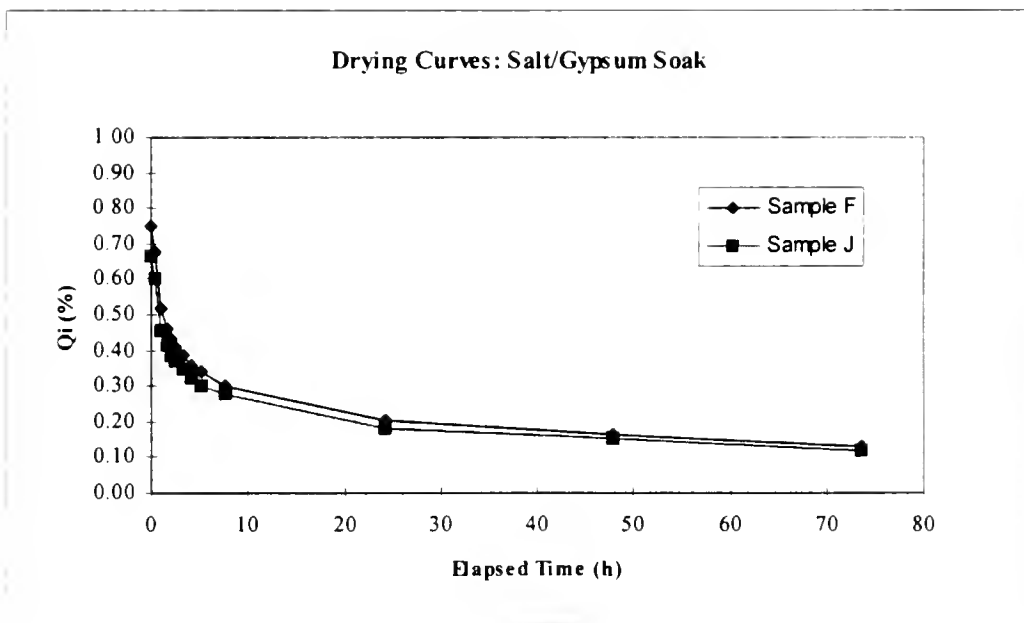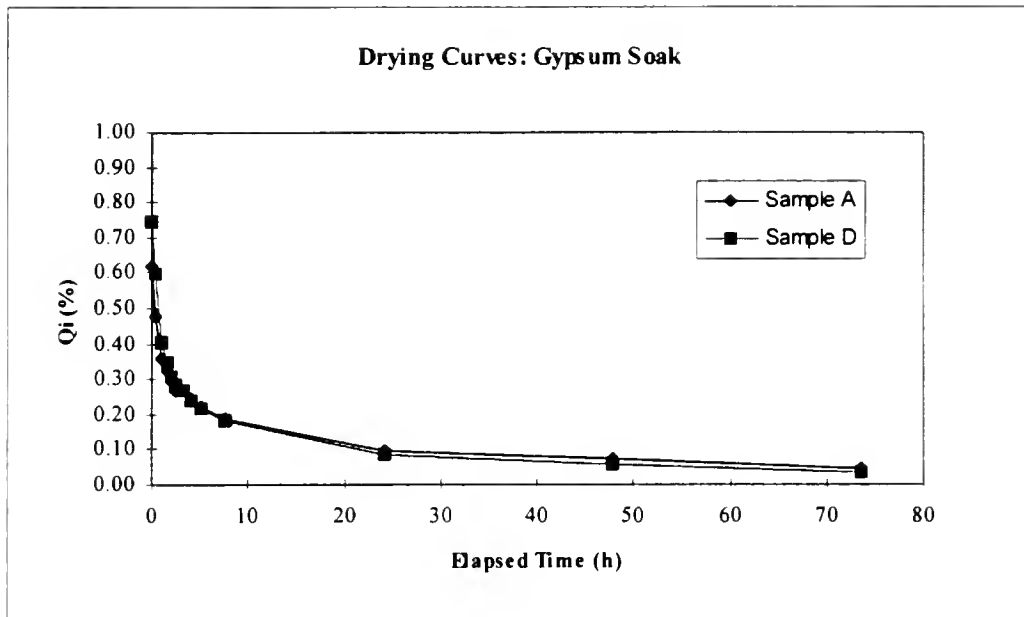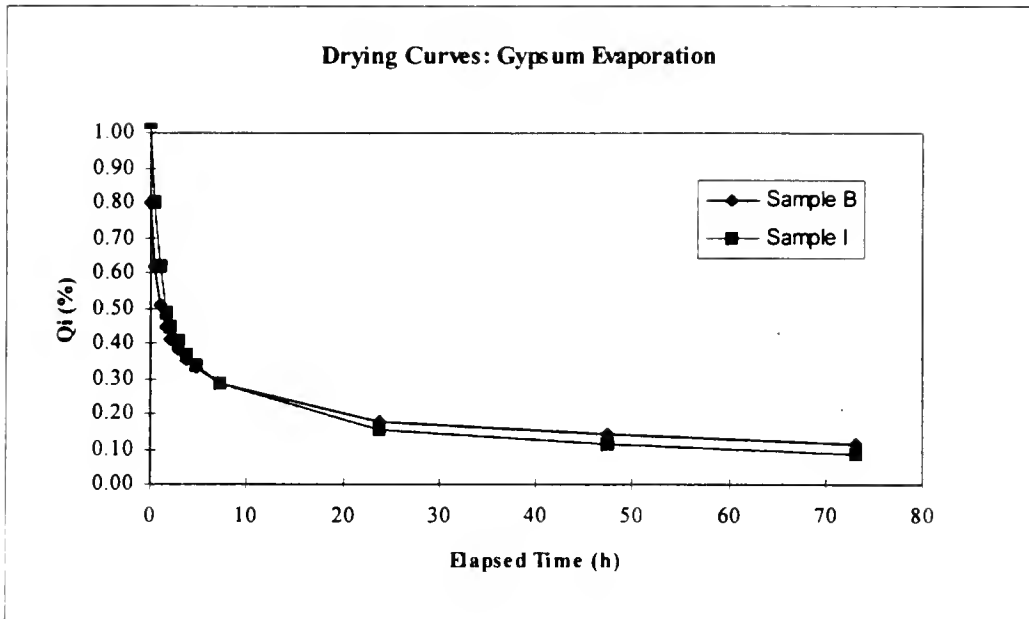
5.4.2   Weight Change

The change in sample weight after weathering is recorded below. It should be noted that the untreated samples lost an average of 0.037%, which should be taken into consideration when evaluating the treated samples. This loss may be due to the degradation of the material itself as a result of interaction with water, to the further removal of residual cutting and cleaning agents. The uncertainty of weighings is ±0.01g.

The greatest gains are seen in the lime- and salt/gypsum-soaked samples, with mean increases of 0.087% and 0.062%, respectively. The gypsum-soaked samples lost a mean

of 0.015%, although this decrease is roughly half of that experienced by the water-soaked

samples and might be considered to be a small relative increase. The gypsum evaporation

samples gained a mean of 0.036%.

Table 5.3: Bulk Sample Weight Change

Treatment	Sample	Pre-Treatment Weight	Treated Weight	% Change
Lime Soak	H	299.53	299.73	0.067
Lime Soak	O	306.34	306.67	0.108
Gypsum Soak	A	300.30	300.24	-0.020
Gypsum Soak	D	313.17	313.14	-0.010
Salt-Gypsum Soak	F	264.45	264.63	0.068
Salt-Gypsum Soak	J	304.38	304.55	0.056
Gypsum Evaporation	B	307.06	307.20	0.046
Gypsum Evaporation	I	264.89	264.96	0.026
Water Soak	C	363.04	362.92	-0.033
Water Soak	E	361.85	361.69	-0.044
Water Soak	K	327.46	327.34	-0.037
Water Soak	P	214.39	214.32	-0.033

5.4.3   Thin Section Analysis

Five thin sections were prepared from bulk samples: one each from the three immersion treatments, one from gypsum evaporation, and one untreated sample. These thin sections taken from the extreme outer surface and were cut perpendicular to the visible foliation. The purpose of these five thin sections was to compare the mechanical changes induced by the four weathering regimens to one another and to untreated stone. The control stone was also compared with the sound stone hand sample.

Thin sections were observed using Polarized Light Microscopy (PLM), and photomicrographs were taken under crossed nicols at 25x magnification. This investigation provided both mineralogical characterization of the material and a qualitative measure of the deterioration states of the hand and bulk samples.

Sample Q (untreated) was used as the control for this examination, and is illustrated in Figures 21 and 22. Some distress is visible both between and within crystals, but the material is generally sound.

Sample G was soaked in a saturated lime solution, and is shown in Figures 23 and 24. The damage to this sample is significant, with extensive microfissures and pores visible

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within the feldspar grains. Notably, this damage extends well into the interior of the stone.

Sample M was soaked in a saturated gypsum solution, and is shown in Figures 25 and 26. Some damage is visible, particularly at the edge of the sample where there is some intercrystalline disruption. Although gypsum is well known to induce damage, this low degree of damage is explained by the extremely low solubility of the salt and the short testing period.

Sample N was soaked in a saturated solution of salt (sodium chloride) and gypsum, and is shown in Figures 27 and 28. This sample is severely damaged, with entire crystals dislodged and intracrystalline fissures visible.

Sample L was used for the gypsum evaporation test, and is shown in Figures 29 and 30. Here the damage is greater than for Sample M, and is particularly visible at the edge. This is consistent with the phenomenon of gypsum crusts and their damaging effects.

Figure 21: Sample Q, Untreated
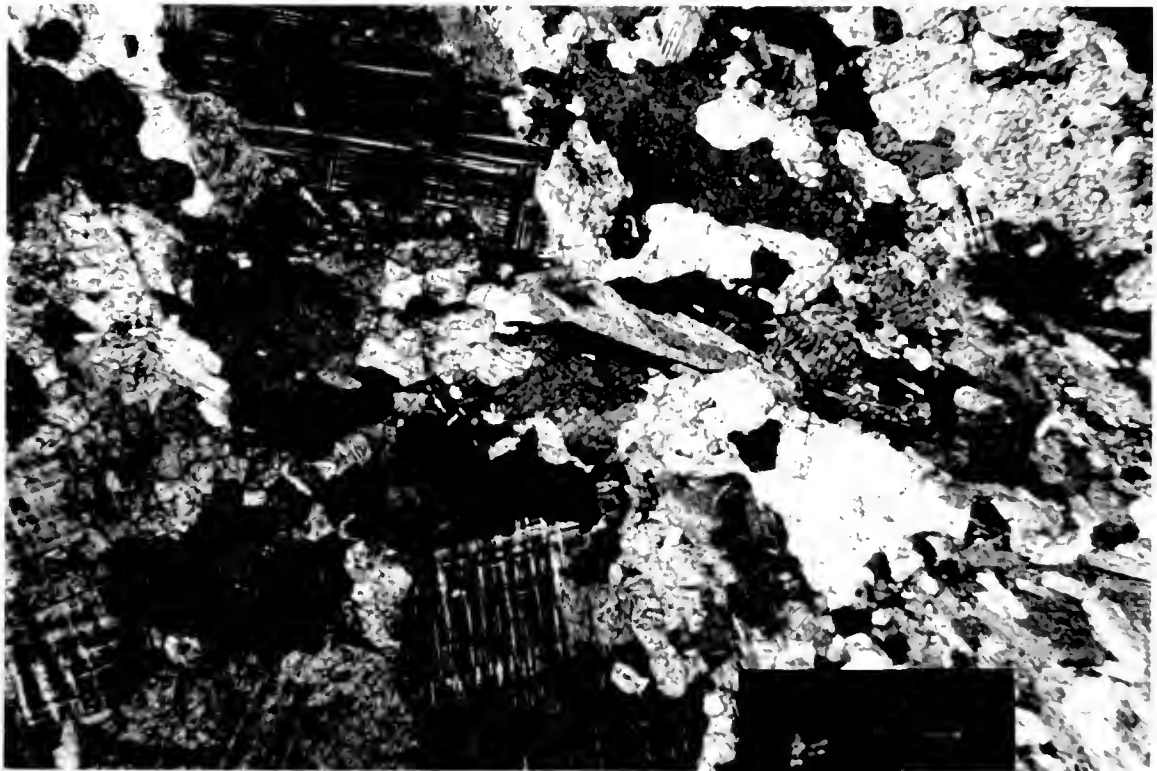
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 22: Sample Q, Untreated
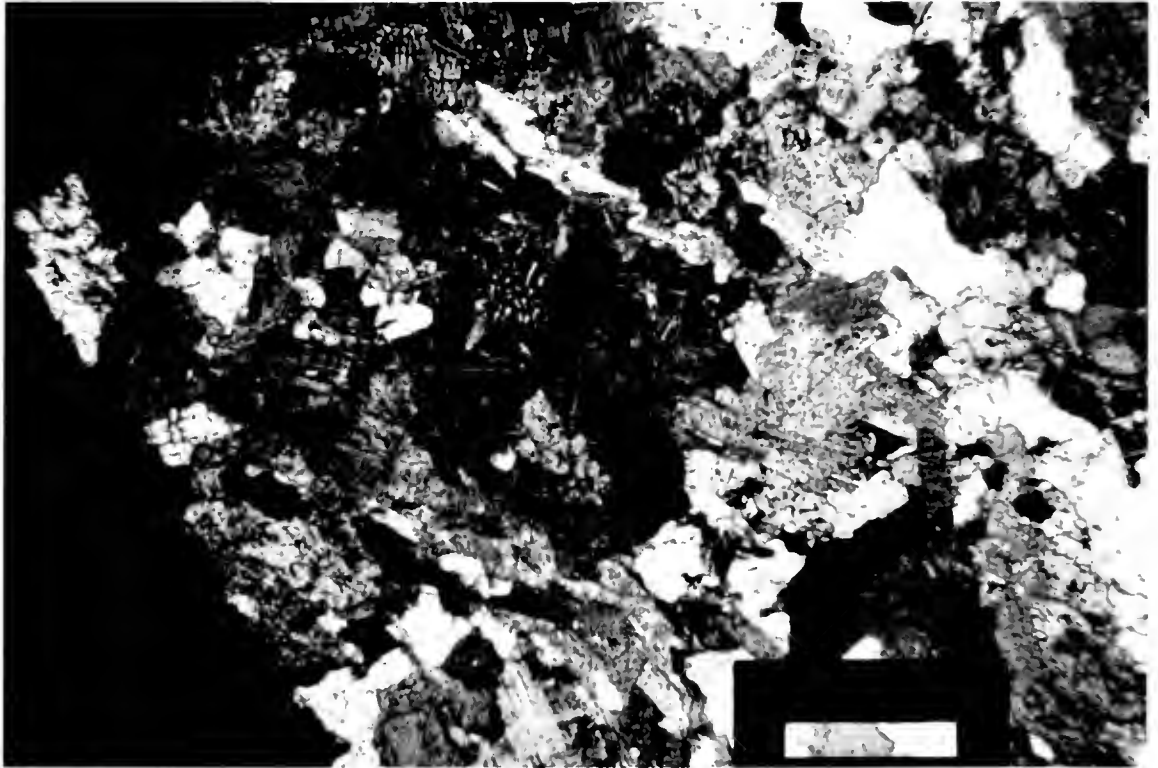
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 23: Sample G, Lime Soak
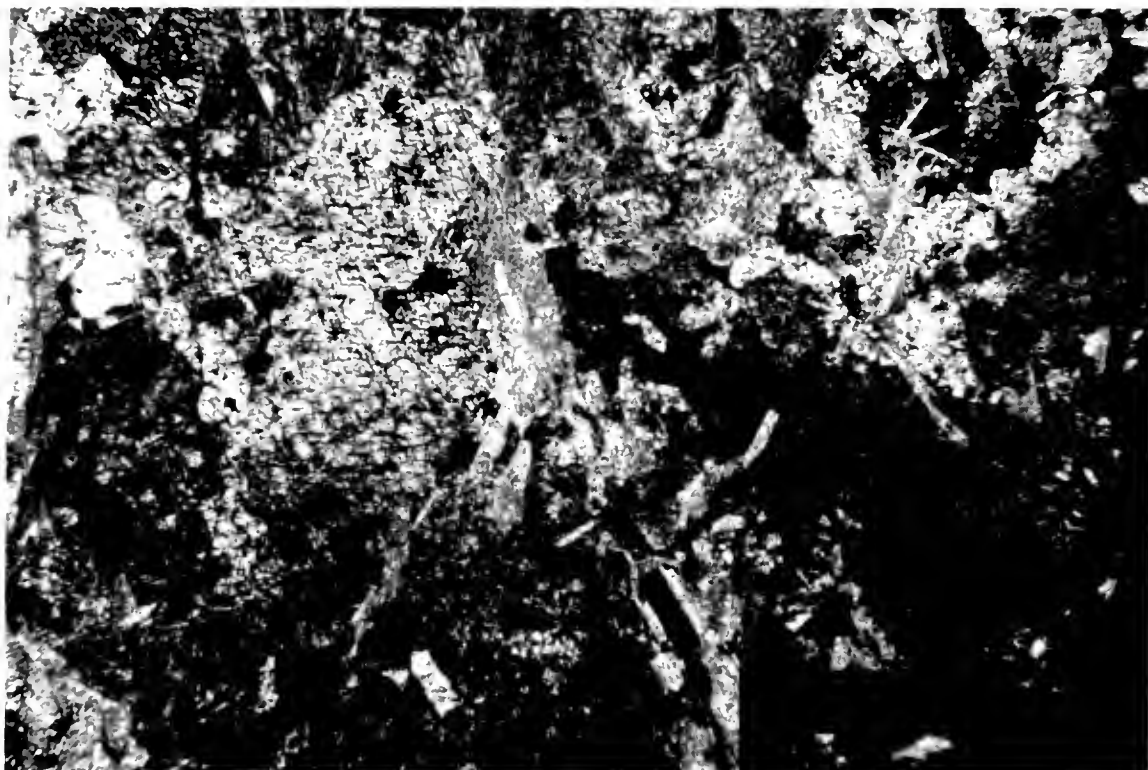
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 24: Sample G, Lime Soak
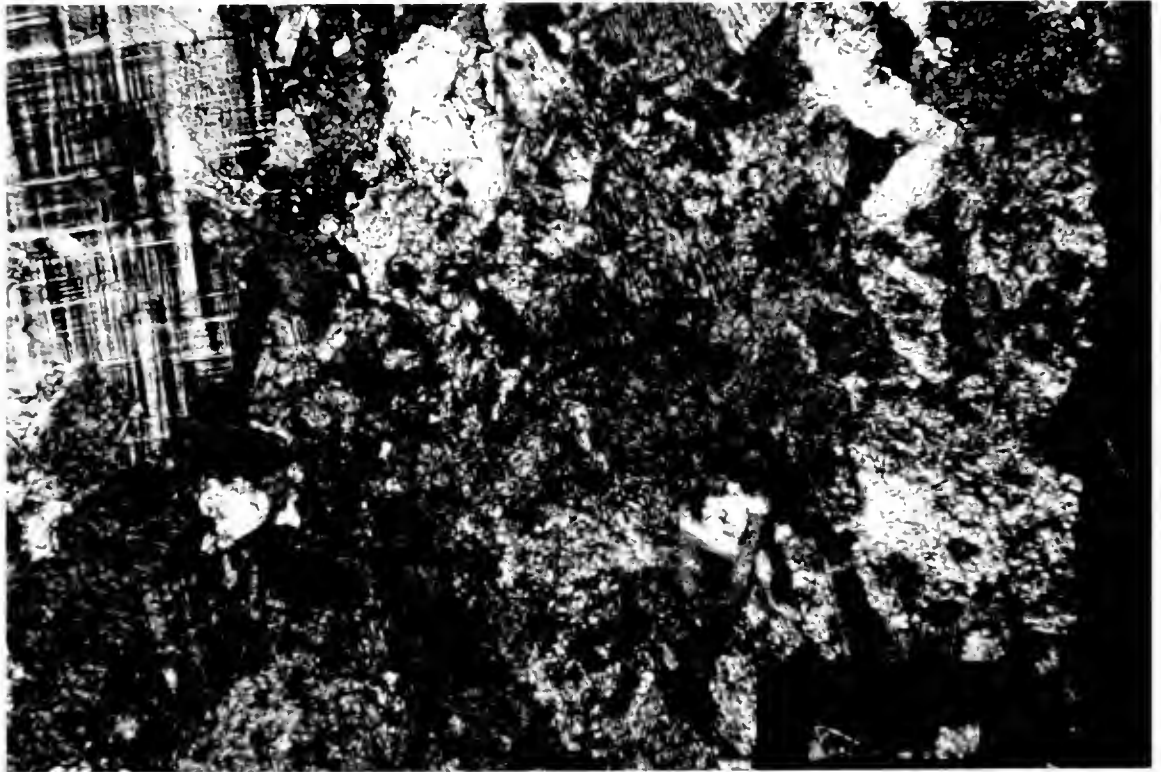
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 25: Sample M, Gypsum Soak
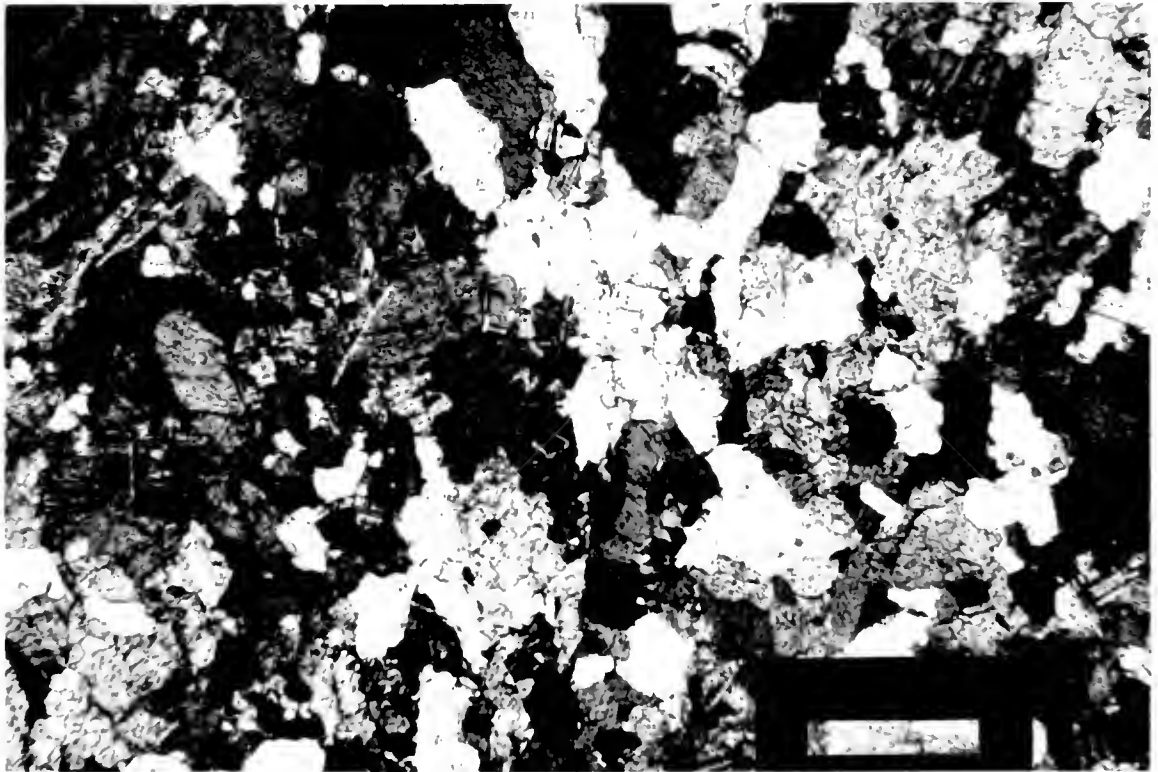
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 26: Sample M, Gypsum Soak
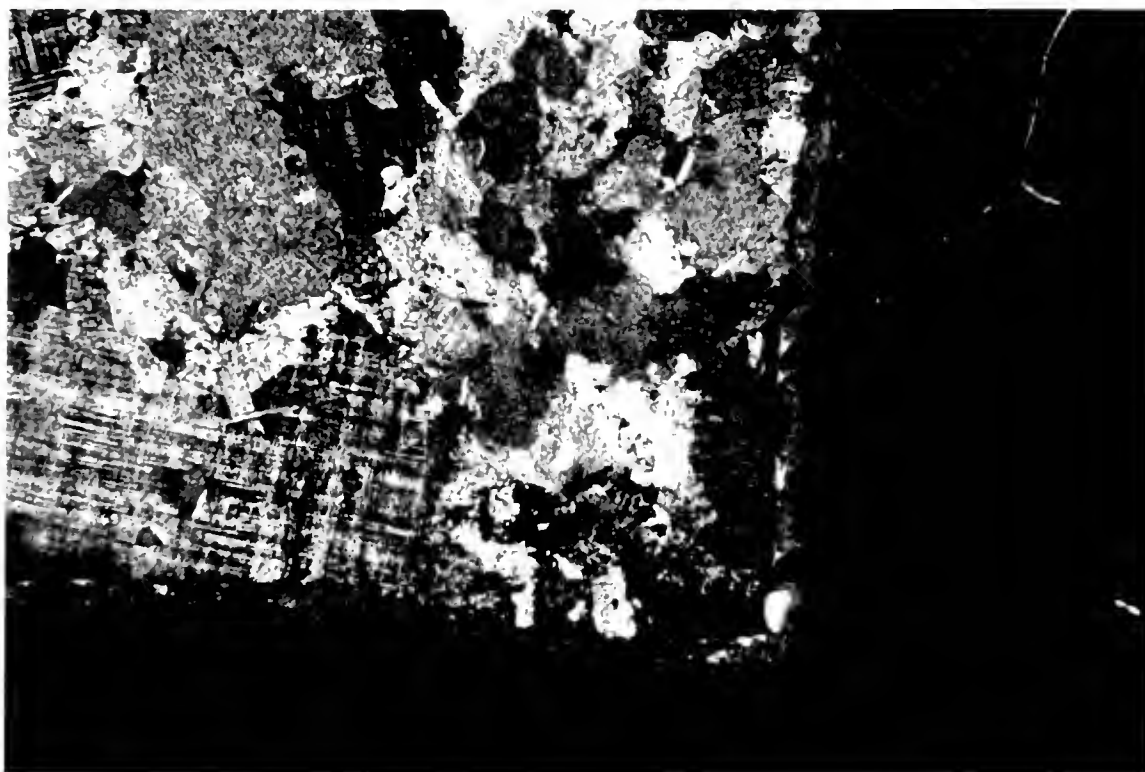
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 27: Sample N, Sodium Chloride-Gypsum Soak
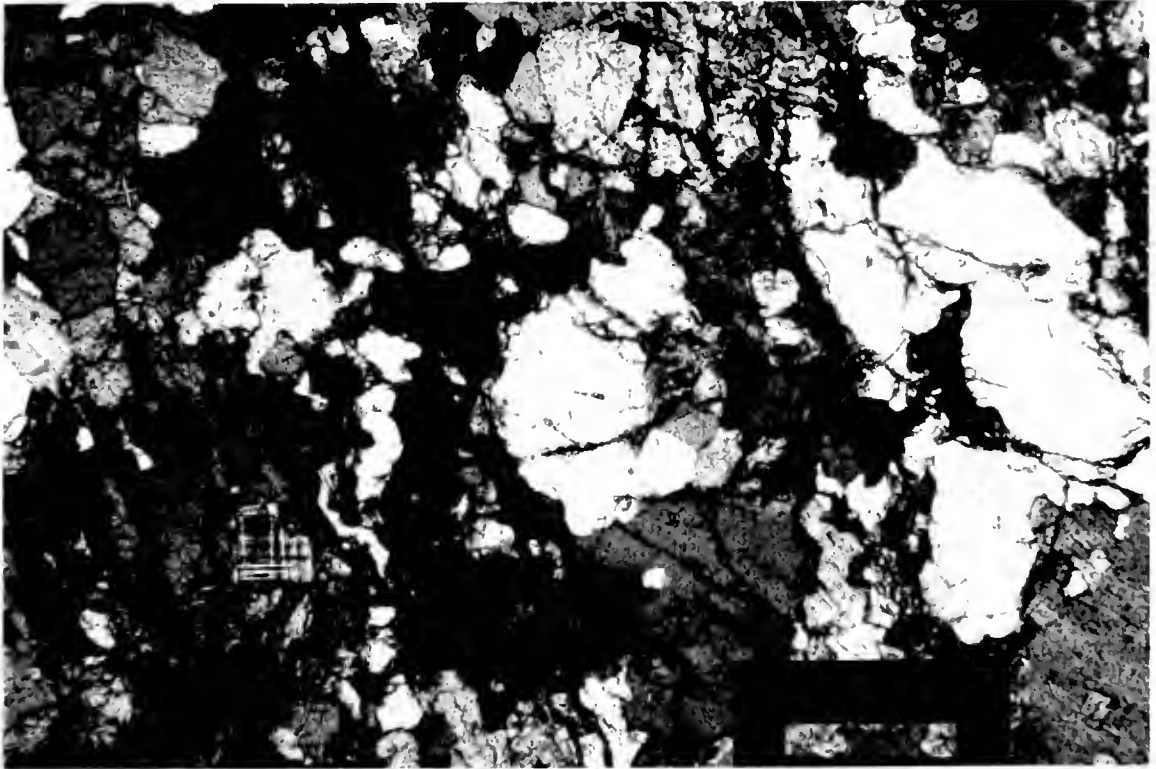
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 28: Sample N, Sodium Chloride-Gypsum Soak
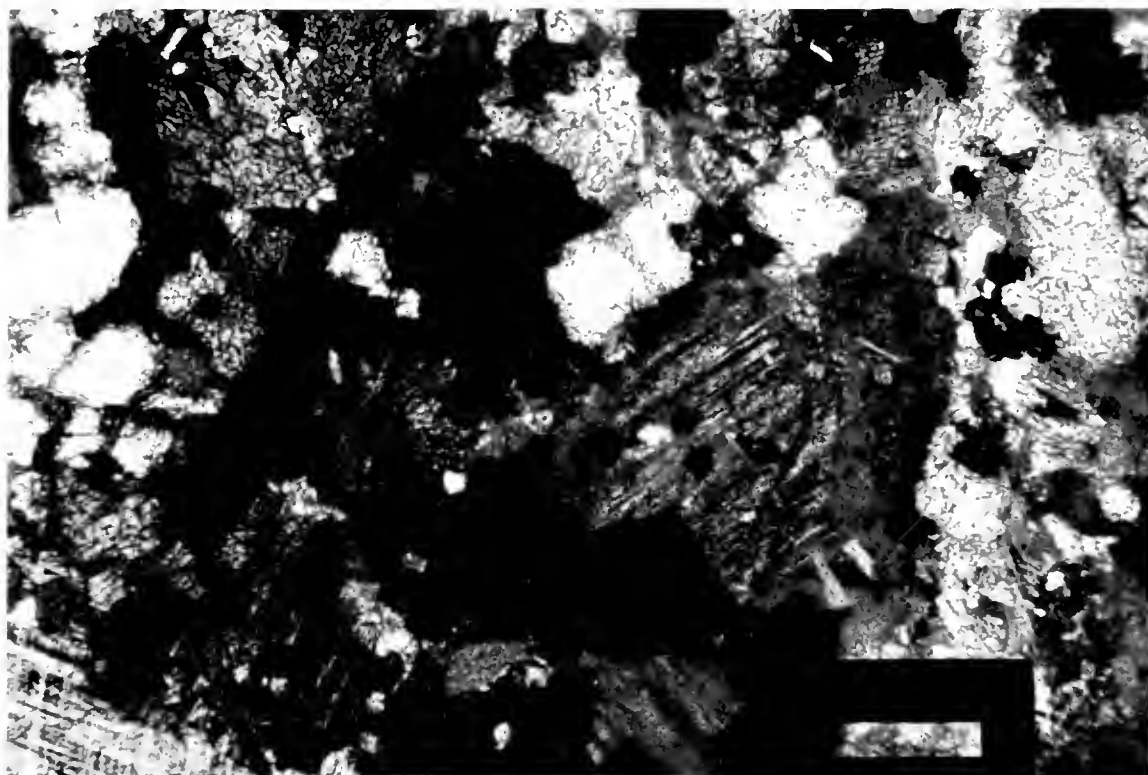
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 29: Sample L, Gypsum Evaporation

Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Figure 30: Sample L, Gypsum Evaporation
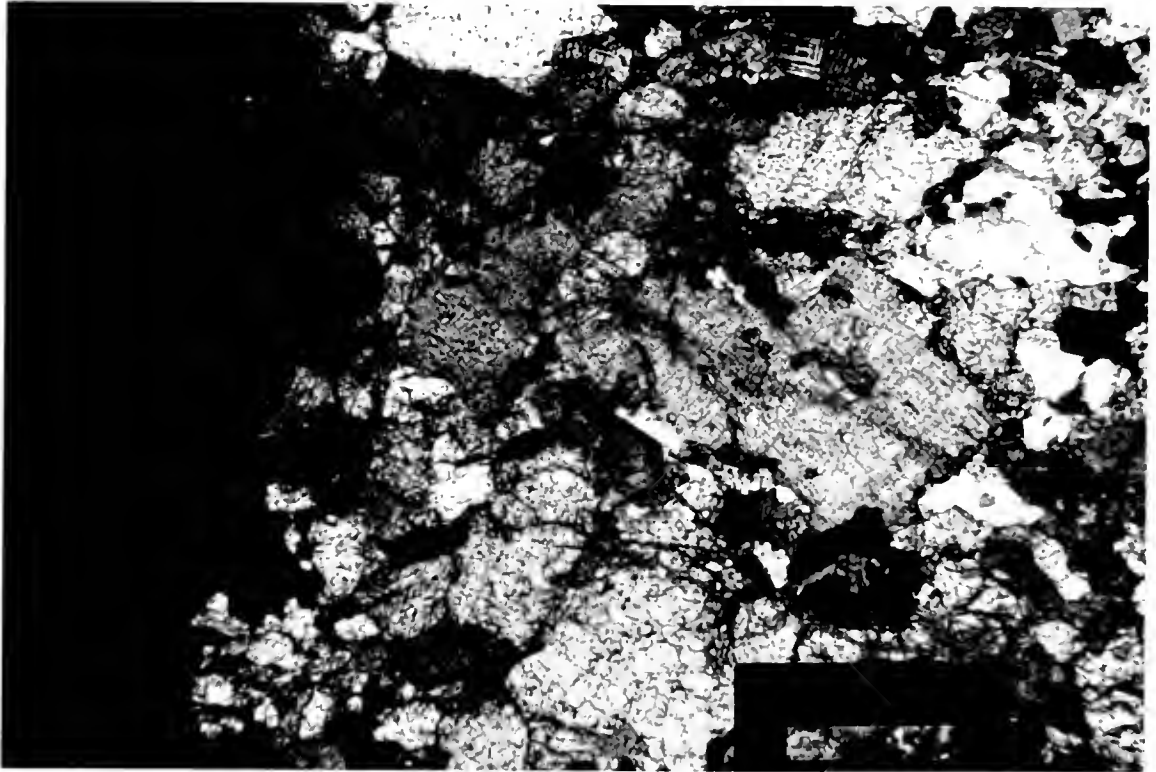
Polarized light photomicrograph under crossed nicols at 25x magnification
Photo: G.I. Omar

Chapter 6: Conclusions

Based on the research conducted in this study, it is clear that the presence of limewash *per se* is damaging to gneiss. The primary mechanism active in this damage appears to be the degradation of feldspars in a strongly alkaline environment due to the incresased solubility of silicates at high pH.[38] [39] Given the high feldspar content of the subject stone, it is not surprising to see this phenomenon active where limewash is applied.

Two secondary mechanisms were also observed: the severe degradation of the gneiss by the sodium chloride-gypsum mixture and the less aggressive but still notable action of gypsum alone. Both of these actions are mechanical, but manifest differently in the tested material.

In the case of the sodium chloride-gypsum solution, the decay is far more aggressive than it is with lime. Intercrystalline disruption is evident, with some damage visible in the affected crystals. As a result of this disruption, the material becomes friable and porous, providing ample access to further water and pollutant infiltration through the damaged

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[38] Drever and Stillings, "The Role of Organic Acids", 167-181.
[39] S.A. Welch and W.J. Ullman, "Feldspar Dissolution in Acidic and Organic Solutions: Compositional and pH Dependence of Dissolution Rate," *Geochimica et Cosmochimica Acta* 60 no. 16 (August 1996): 2939-2948.

surface. In the sodium chloride-gypsum solution, the solubility of gypsum is increased, resulting in even greater damage upon its crystallization than in the case of gypsum alone.

The damage induced by gypsum, although less severe than that of the other materials in the time covered by this testing program, is visible here and is well known and documented in the conservation literature. As mentioned above, the mechanism of gypsum deterioration is mechanical, due to the recrystallization of the salt from solution, which will dislodge particles of the affected stone. In this testing program, such mechanical damage is evident to some degree in all of the gypsum-treated samples, and to an even greater extent in the presence of sodium chloride.

The decay morphology is different in all cases, and particularly notably for the two most damaging solutions, lime and sodium chloride-gypsum. The overall effect of the decay due to lime is one of numerous small fissures and pores both within and between crystals, with most crystals being affected to a similar degree. In contrast, the effect of sodium chloride is one of more wholesale disruption of the crystalline structure. Intracrystalline damage in the remaining material generally appears at a larger scale than is visible with lime, although some microcracking is evident in these samples as well. Moreover, since sodium chloride is hygroscopic at high humidities, the increased presence of water within

the material will increase the chemical activity and any working dissolution and recrystallization mechanisms.

As all three tested materials are damaging to gneiss, we may conclude that any combination of lime-based render or mortar, sodium chloride from any source, and Portland cement render or mortar will be extremely damaging to this stone. Although granitic rocks are generally considered durable and excellent building material, as with any material, care must be taken to assure its compatibility with other elements of the building system.

As the definition of historically significant buildings expands to include not only great cultural and religious monuments, but the structures of everyday life, the materials upon which the conservation community will focus its efforts will perforce change to meet the needs of this new patrimony. Similarly, we may expect the demand for information on local materials to increase. It is hoped that this study has helped to elucidate some of the mechanisms of deterioration of gneiss in the presence of alkaline materials and to suggest further avenues of investigation in this area.

Appendix A

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Appendix B

Decay Classifications

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1. GRANULAR DISINTEGRATION

Material loss by the progressive detachment of single grains or grain aggregates causing an advanced state of surface incoherence, a considerable decline in the original mechanical strength of the material and an increase in porosity. The quantitative physical description of granular disintegration includes the following characteristic: frequency of occurrence per unit area -- slight, medium, severe.

2. BIOGENIC GROWTH

Macroscopic overgrowth of living organisms and/or colonization by microflora, causing deterioration and in more severe cases structural disruption in the form of: fungi, bacteria, algae (endolithic, epilithic) which may show various colors besides green, may be white, pink, or yellow; lichen (crustose, planar); ferns and mosses; higher plants -- trees, climbers and creepers; small flowering plants; birds and bees. The quantitative physical description of biogenic growth includes the following characteristic: frequency of occurrence per unit area -- slight, medium, severe.

3. BLISTER

Swelling and superficial lifting of the surface and detachment of small planar grain aggregates of variable shape, color and firmness causes surface disruption which leads to flaking and scaling. The quantitative physical description of blistering includes the following characteristics: size and frequency of occurrence per unit area -- slight, medium, severe.

4. FLAKES

The parallel detachment of small, thin, planar grain aggregates in the form of flakes from the surface or the base substrate. The quantitative physical description of flaking includes the following characteristics: size -- from 5mm - 15mm; and frequency of occurrence per unit area -- slight, medium, severe.

5. SCALES

The lifting, followed by the detachment of large, planar elements, of one or more thin surface layers which are parallel to one another. The scales are usually of uniform thickness and are composed of either intact or altered material. The quantitative physical description of scales includes the following characteristics: size -- from 15mm - 30mm; and frequency of occurrence per unit area -- slight, medium, severe.

6. RUPTURE

A loss of compact stone fragments mostly due to direct anthropogenic influence or other mechanical influence. The quantitative physical description of rupture includes the following characteristic: size -- from >30mm.

7. SPALLING

A deterioration evidenced by the total or partial detachment and loss of more or less isometric grain aggregates, chips or splinters, or solid stone pieces often following discontinuity of planes in the original material. The chips or splinters are irregular in form and thickness, and vary in size. This form of deterioration is weather related. The quantitative physical description of spalling includes the following characteristic: size -- from >30mm.

8. STAINING

The deposition of external particles on the surface, partly loosely fixed, consisting of a flat hard layer which usually covers large areas and is found in areas significantly well sheltered. this damage type is characterized by a change in color (hue) or in color intensity (saturation) usually in the form of black staining. Other colors (red, green, brown), orientation (may include vertical, horizontal), pattern (streaks, spots, liesgang rings), associations (pyrites, tarry pollutants, rising damp, mortars containing hydraulic lime or Portland cement, copper and its alloys, iron and steel). The quantitative physical description of staining includes the following characteristic: frequency of occurrence per unit area -- light, medium, severe.

9. CRUSTIFICATION

A compact, three-dimensional, framboidal, layered growth of rather insoluble compounds, frequently carbonates attached to the surface. The deposit can be distinguished visually from the underlying parts by its morphological characteristics and often by its color. The crust and underlying stone are stable and deposited on the surface by water, then the recrystallization of calcium carbonate of the substrate occurs, dissolved and reprecipitated from water rich in carbon dioxide. Colored by an accumulation of foreign material e.g. dust, dirt, soot, pigeon droppings, micro-organisms.   The

quantitative physical description of crustification includes the following characteristic: frequency of occurrence per unit area -- light, medium, severe.

10. SALT EFFLORESCENCE

Loose deposits of salt aggregate crystal crusts on surface, or below flakes and scales -- associated with the presence of generally white coatings of soluble salts; salts may also be in the stone, mortar or other substrate. Color: translucent, bright white and crystallization: frequently incoherent. The quantitative physical description of salt efflorescence includes the following characteristic: frequency of occurrence per unit area -- light, medium, severe.

11. CRACKING

The phenomenon due to mechanical stress and weathering is manifested by a break extending through to the surface. Where this is difficult to determine, the break should be called a crack only if the underlying surface is visible. Three types of cracking are recognized:

1. Individual: determined by length, width, and orientation;
2. Line Type (bed separation): cracking in which the breaks are arranged in parallel lines, usually either horizontally or vertically over the surface, or failure of the stone due to inappropriate design; and
3. Sigsmoid Type/Networks (dendritic, parallel, trellis, rectangular, radial, annular): cracking in which the breaks form a pattern consisting of curves meeting and intersecting usually on a relatively large scale.

The quantitative physical description of cracking includes the following characteristic: frequency of occurrence per unit area -- light, medium, severe.

12. RELIEF

Morphological change of stone due to the partial or selective weathering out of bedding planes and 'soft' seams or pockets. A loss of material occurs due to wind-driven particles in the form of alveolar formation, honeycombs, severe pitting/alveolar erosion and occasional chromatic alteration. The quantitative physical description of relief includes the following characteristic: frequency of occurrence per unit area -- light, medium, severe.

Appendix C

Testing Protocols

C.1 NORMAL 11/85: Capillary Water Absorption and Capillary Absorption Coefficient

C.2 NORMAL 29/88: Measurement of the Drying Index

C.3 Qualitative Salt Analysis

NORMAL 11/85   *DRAFT TRANSLATION*

CAPILLARY WATER ABSORPTION AND

CAPILLARY ABSORPTION COEFFICIENT

Group F   Subgroup F/1

OBJECTIVE   Capillary Water Absorption - Capillary Absorption Coefficient

FIELD OF APPLICATION   Stone Materials: sound and deteriorated, treated and untreated

SCOPE   ·   Characterization of stone materials and evaluation of its deterioration or of the effect of treatments

Preface

This second edition of the document cancels the previous one. It adapts the described methodology for the case of stone samples treated with products which modify their water-repellency characteristics.

1   DEFINITION OF THE MEASURED PROPERTY

Capillary water absorption is the amount of water absorbed per unit surface $[g/cm^2]$ as a function of time, t, at room temperature and pressure, by a sample which has its support surface in contact with de-ionized water.

Capillary absorption coefficient: angular coefficient, expressed in $g/cm^2.s^{1/2}$, of the initial straight segment of the capillary absorption curve.

2   SAMPLE PREPARATION

2.1 Shape and Sizes

The samples must have a regular shape: cubes, cylinders or parallelepipeds. The ratio of surface to apparent volume must be:

$$1 \text{ cm}^{-1} \leq s(cm^2)/v(cm^3) \leq 2 \text{ cm}^{-1}$$

NORMAL 11/85

For example, the edge of cubes must be between 3 to 5 cm. All samples in each series must have the same shape and size. The minimum dimension of the sample must be compatible with the homogeneity of the stone material under examination.

After cutting the samples must be washed with de-ionized water, and if necessary brushed with a soft-bristled brush, to remove any powder left on the surface.

2.2 Drying Procedure
Each sample is dried in a forced ventilation over at 60°C ± 5°C and cooled in a desiccator prior to weighing.

The 60°C temperature is chosen to avoid alteration of any product that may have been applied in a conservation treatment.

The drying procedure is repeated until constant weight is attained. This is achieved when the difference between two successive weighings at 24 hour difference is $\leq$ 0.1% of the weight of the sample.

2.3 Number of Samples
The measurement must be carried out on a number of samples which depends on the heterogeneity of the stone material. However, each set must at least consist of three samples. The samples in each series must be comparable in homogeneity and degree of alteration.

3   MEASUREMENT PROCEDURE

Each sample, after drying and weighing, is set into a container on a porous support constituted by a pack of circular filter paper of the fast-filtering type Schleicher & Schüll n° 589 black band or Whatman n° 4 (?) or 41.

Should the measurements be carried out on treated samples, care must be taken that the treated surface be used as the absorbing face and put in contact with the filter paper.

The filter papers mentioned have an average pore diamente of 25 μm; the thickness of the pack should be of about 1 cm. The use of the filter papers ensures an immediate and constant contact between the water and the support (absorbing) surface of the sample, even in the case of samples with rough surfaces or not perfectly regular.

De-ionized water is poured in slowly until the filter paper is totally wetted but keeping the water level below the top surface of the filter paper pack. To reduce the evaporation of the water and the influence of any changes in the thermohygrometric ambient conditions, the container must be provided with a lid. This can result in condensation of water vapour in the interior walls of the container. To avoid the fall of water drops onto the samples the cover can be lined with a piece of filter paper. Furthermore, the container can be protected with a reflecting cover, such as aluminum foil, to prevent irradiation and consequent moisture condensation on the samples.

NORMAL 11/85

At given time intervals the samples are weighed: they are taken out of the container and the wet surface patted dry with a damp cloth; after weighing they are put back into the container. The test is continued until the variation in the amount of absorbed water in two successive weighings, at a 24-hour interval, is $\leq$ 1% of the amount of water absorbed.

The experimental values obtained are plotted in a "capillary absorption curve", i.e., the amount of water absorbed per unit surface as a function of $\sqrt{t}$.

In general the curve has a variable beginning depending on the condition of the sample (treated or untreated) but then tends asymptotically to reach a constant value of absorbed water. It is necessary to clearly define the linear initial portion to be able to obtain the tangent (see 4.3), therefore at least three experimental points must be obtained in the linear segment of the curve. The choice of the optimum time intervals for the initial period is made depending on the absorption characterisitcs of the samples and must be made on one or two preliminary samples before the actual samples in a series are measured. In those cases in which the time intervals are very small ($\leq$ 5 minutes) the error introduced by following the described procedure would be too large. In these instances, after each weighing the sample is drying again as described in section 2.2, before returning the sample to the container and taking the following measurement. Regardless of the chosen initial time intervals, weighings have to be made at 1 h, 8 h, 24 h of capillary absorption and repeated until the asymtotical value is reached (see 4.2).

4   DATA ELABORATION

4.1 Calculation of the Capillary Water Absorption

The amount of water absorbed by the sample per unit surface (M_i) at a time t_i, is calculater with the following equation

$$Mi = [m_i - m_o]/S$$

where: m_i = weight of the sample at time t_i (g);

m_o = weight of the dry sample (g);

S = surface of the sample in contact with the porous support (cm^2) given with a 5% precision.

The average values M_i for each series is calculated and plotted in a graph as a function of $\sqrt{t}$, where t is given in seconds. In general, the shape of the curves is similar to those illustrated in Appendix 1, Graph B.

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4.2 Evaluation of the Asymtotical Value

The asymtotical value M^* can be considered as reached in a capillary water absorption curve when the difference in weight ΔM of the amount of water absorbed between two successive points is in the order of 1%. At this point:

$$Mi = M^*$$

when

$$DM = [(M_i - M_{i-1})/ M_i]\ 100 \leq 1\%$$

where

M^* = aymptotical value of the amount of absorbed water per unit surface of the sample $[g/cm^2]$;

M_i and M_{i-1} = amount of water absorbed per unit surface of the sample at times t_i and t_{-1} $[g/cm^2]$.

The asymptotical value may not be reached within reasonable times and in this case it is convenient to stop the test before secondary phenomena, such as the growth of microorganisms, occur. In these cases it is evidently impossible to calculate the asympotitcal value M^* and an approximation can be obtained calculating the average value of the amount of water absorbed M_i between 10 and 11 days.

4.3 Calculation of the Capillary Water Absorption Coefficient

The capillary absorption coefficient AC is represented by the tangent of the linear segment of the capillary absorption curve. It can therefore be calculated as the ratio between the ordinate M and the absissa $\sqrt{t}$. For untreated specimens the calculations can be carried out applied the equation:

$$AC = M^*/\sqrt{t^*}\quad [g/cm^2.s^{1/2}]$$

where

M^* = asymptotical value of the amount of water absorbed by the sample per unit surface $[g/cm^2]$;

t^* = abscissa at the intersection point of the line extrapolated from the asymptote and the tangent of the straigt segment of the curve $[s^{1/2}]$.

4.4 Errors

The maximum deviation from the mean value of the amount of absorbed water M_i is taken as the error of that value.

NORMAL 11/85

5   RESULT PRESENTATION

The report of the analysis must present the experimental results obtained in a:

a) Table which lists

- number, shape and size of the samples;
- the average values of the amount of absorbed water $\overline{M_i}$ per unit surface and the corresponding times;

- the asymptotical value of the amount of absorbed water M^*, per unit surface, or if this is unobtainable, the average values of absorbed water per unit surface $\overline{M_i}$ measured at 10 and 11 days.

- the water absorption coefficient AC;

b) a graph of the $\overline{M_i}$ values as a function of $\sqrt{t}$.

Appendix I gives an example of a Table A and the corresponding Graph B for a generic series of samples.

A   TABELLA ESEMPLIFICATIVA RELATIVA AD UNA GENERICA SERIE DI CAMPIONI

Tempo √s	M/S (10^{-1} g/cm^2)		
	Campione non trattato	Trattamento a)	Trattamento b)
30.0	1 48	0.04	0.24
42.4	1.89	0.06	0.27
60.0	2.40	0.09	0.27
84.8	2.84	0.15	0.39
103.9	3.03	0.19	0.42
120.0	3.04	0.26	0.49
169.7	3.13	0.58	0 60
293.9	3.25	1.39	0 64
415.7	3.22	1.77	0.69
509.1	3 44	2.11	0 69
587.9	3.32	2.09	0.70
657.3	3.33	2.14	0.76

B   GRAFICO ESEMPLIFICATIVO RELATIVO AD UNA GENERICA SERIE DI CAMPIONI
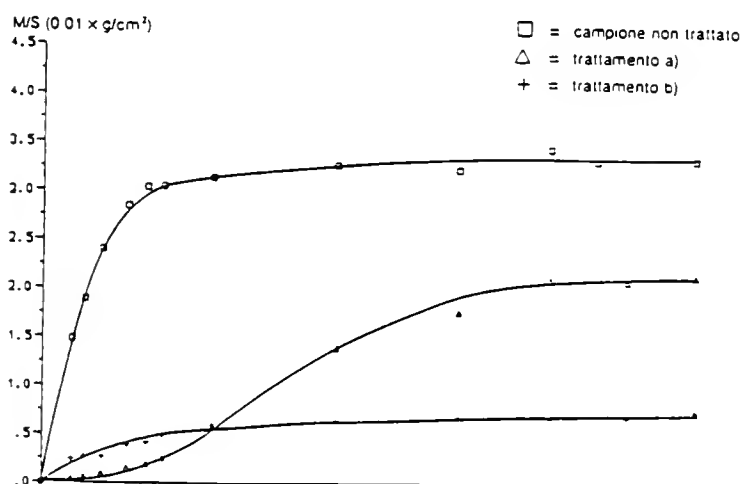
NORMAL 29/88

NORMAL 29/88   *DRAFT TRANSLATION*

MEASUREMENT OF THE DRYING INDEX

Group F   Subgroup F/1

OBJECTIVE   Measurement of the loss by evaporation of the water absorbed by the material

FIELD OF APPLICATION   Stone materials, sound and deteriorated, treated and untreated

SCOPE   Characterization of the stone material, evaluation of the deterioration and of the effects of conservation treatments

Preface

The measurement is carried out on samples saturated with water as obtained in the water absorption by total immersion test (NORMAL 7/81)

1   DEFINITION OF THE MEASURED PROPERTY

The variation of water content of the material over time, at constant temperature and relative humidity, is measured and expressed as a percentage of the dry weight of the sample.

Drying Index: is the ratio between the integral of the drying curve and the maximum water content multiplied by the final time, i.e., the time required in drying and taken as that reached asymptotically by the curve.

2   PREPARATION OF THE SAMPLE

This follows the procedure described in sections 2 and 3 of NORMAL 7/81.

3   MEASUREMENT PROCEDURE

The sample saturated with water (NORMAL 7/88 section 3) is weighed after patting it dry with a damp chamois cloth to avoid the presence of excess water on the surface. Immediately after the weighing each sample is set upon a rigid net of non-corrodable material (net opening of 1 x 1

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cm) inside a desiccator supplied with silica with an indicator (cobalt chloride). This desiccator must be kept in a constant temperature (20°C ± 1°C) room.

At given time intervals the sample is taken out of the desiccator and weighed.

The choice of weighing times during the first 24 hours must be made according to the evaporation characteristics of the material and on the basis of a preliminary test so as to identify the time frame of maximum variation as well as the initial evaporation rate.
After this period, the weighings are repeated at 24-hour intervals, when the following formula applies:

$$1.0 \geqslant \frac{m_0 - m_{i-1}}{m_0 - m_i} \geqslant 0.90$$

where: m_0 = weight [g] of the sample at time t_0 [h];

m_{i-1} = weight [g] of the sample at time t_{i-1} [h];

m_i = weight [g[of the sample at time t_i [h].

After this the sample is dried in an oven at 60°C ± 5°C, until constant weight. The weight is considered cosntant when the differences between two weighings at 24-hour difference are smaller or equal to 0.01% of the weight of the dry sample.

With the experimental values obtained a "drying curve" is plotted (amount of water as a function of time, see section 4).

The size of the desiccator and the number of samples that can be put into each desiccator must be chosen, by preliminary testing, as a function of the nature of the stone material and to ensure that the relative humidity within the desiccator does not increase above the equilibrium value for the anhydrous silica gel.

The control of the relative humidity within the desiccator is carried out by having an indicador leaning against one of the desiccator walls. As indicators the commercial cobalt chloride strips may be used, or these may be prepared by imbibing filter paper strips with a 10% aqueous solution of cobalt chloride.

In the case of very porous samples it is necessary to include, in the desiccator, between the samples silica gel bags made out of a permeable material.
During the length of the test the indicator must keep its blue colour. Otherwise, the silica gel in the bottom of the container must be rapidly mixed, or even changed, or extra silica gel bags added or the number of samples reduced.

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4   DATA ELABORATION

4.1 Calculations

a) the residual water content Q_i of the sample at time t_i is calculated with the following equation:

$$Q_i = \frac{m_i - m_d}{m_d} \cdot 100$$

where Q_i = water content at time t_i, given as percent of the final dry weight;

   m_i = weight [g] of the sample at time t_i [h];

   m_d = weigh [g] of the dry sample at the end of the test.

The values of Q_i are plotted in a graph as a function of time t_i.

The initial maximum water content (Q_{max}) corresponds to the water imbibition capacity (IC) determined during the total immersion procedure (see NORMAL 7/81).

b) drying index. The drying index (DI) is calculated with the equation:

$$DI = \frac{\int_{t_o}^{t_f} f(Q_i)\, dt}{Q_{max} \cdot t_f}$$

where

$f(Q_i)$ = water content (expressed as percentage of the final dry weight) as a function of time;

Q_{max} = IC = initial water content, expressed as percentage of the final dry weight;

t_f = final time of the test, given in hours;

t_o = initial time of the test = 0

The calculation of the integral of the curve can be done following the Simpson method:

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$$\frac{t_f - t_o}{3n} \left[Q_{max} + Q_f + 2(Q_2 + Q_4 + \dots + Q_{n-2}) + 4(Q_1 + Q_3 + \dots + Q_{n-1}) \right]$$

where

Q_{max} = initial water content at time t_o, expressed as percentage of the final dry weight;

Q_f = final water content at final time t_f, expressed as percentage of the final dry weight;

t_o = initial time of the test = 0;

n = number of intervals established for the calculation;

t_f = final time of the test.

The final time of the test is considered the time at which the curve reaches asymptotically the abscisa. In the evaporation curve this can be considered the time when the equation (1) is verified.

When different materials are to be compared, the calculation of the drying index must take the same final time for all of them.

For comparison between materials before and after treatment, the final time is taken as that corresponding to the samples prior to treatment.

The number of intervals is chosen by successive approximations so as to satisfy the equation:

$$\frac{(DI)_n - (DI)_{n-1}}{(DI)_n} \times 100 \leq 5$$

where $(DI)_n$ = drying index calculated for n intervals;

$(DI)_{n-1}$ = drying index calculated for n-1 intervals.

5   RESULTS PRESENTATION

The report of the analysis must include:

a) a table where the following information is given.
- shape and size of the sample;
- experimental values of the water content Q_i and the corresponding t_i;
- initial water content, Q_{max}, expressed as percentage of the final dry weight;
- final time of the test, t_f;
- Drying Index (DI) @ t_f;
- the calculation method used for the integral and the number of intervals used.

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b) a graph of the plot of Q_i as a function of time.

In Appendix I are reported examples of a table (A) and a graph (B) relative to a generic series of samples.

APPENDIX I

(A) Example of a Table

Results of the Analysis

Shape of the samples: cubes
Size of the samples: 5-cm side
Number of samples: 5

Q_{max} = 13.49%
t_o = 0
t_i = 24
$(DI)_{24}$ = 0.20

Method used for the calculation of the integral: Simpson's method.
Intervals used: 24

t_i [h]	Q_i (%)
0	13.49
0.50	12.57
1.00	11.72
3.00	8.43
6.00	3.76
9.00	2.15
24.00	0.20

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(B) Graph illustrating the water content Q_i as a function of time.

B   GRAFICO ILLUSTRATIVO DEL CONTENUTO D'ACCUA Q_i IN FUNZIONE DEL TEMPO

Ex. 16 (continued)

d) Another possible origin of sulfates is microbiological. In brief, there are certain types of micro-organisms capable of metabolizing reduced forms of sulfur and oxidizing it to sulfates, as well as others which produce sulfides instead. These "sulfur bacteria" are often present on exposed stone, especially calcareous types. Since there is a strict analogy between calcareous stones and mortars based on calcium carbonate, it is logical to assume that these bacteria could also develop in plasters and mortars

e) The most important source of sulfates, however, is atmospheric pollution. The burning of hydrocarbons leads to the transformation of the sulfur they contain into sulfur dioxide (SO_2), emitted into the atmosphere as a gas

Reacting with oxygen (O_2), sulfur dioxide (SO_2) becomes sulfur trioxide (or sulfuric anhydride, SO_3).

This last product (SO_3) reacts with water (H_2O) to form sulfuric acid ($SO_3 + H_2O = H_2SO_4$) which attacks the calcium carbonate ($CaCO_3$) and transforms it into calcium sulfate ($CaSO_4$).

All these processes can happen in the wall. Alternately, the sulfuric acid can form in the air and then react with the calcium carbonate ($CaCO_3$) of the wall.

A third possibility is that the sulfuric acid formed in the air is neutralized by basic substances such as ammonia (NH_3), forming ammonium sulfate ($(NH_4)_2SO_4$).

or by calcium carbonate present as atmospheric dust, forming calcium sulfate (gypsum, $CaSO_4$). In a polluted environment, these sulfate solids can constitute 20 to 30% of the dust in the atmosphere.

As a final possibility, studies have shown that sulfur dioxide (SO_2) can be absorbed directly or as sulfurous acid (H_2SO_3). This leads to the formation of calcium sulfite ($CaSO_3 \cdot 2H_2O$) which oxidizes to sulfate.

The processes described above are summarized in the chart on the following page:

Ex. 16 QUALITATIVE ANALYSIS OF WATER-SOLUBLE SALTS AND CARBONATES

AIM/INTRODUCTION

The presence of white efflorescence on the surface of masonry is always an indication of chemical deterioration processes resulting from the reaction of three components: the materials themselves, water, and polluting compounds present in the water or the atmosphere. (A fourth component can be contributed by micro-organisms.)

The deterioration products resulting from the reaction between these three components are water-soluble salts, principally sulfates, chlorides, nitrites and nitrates. Under certain conditions, calcium carbonate (a normal component of mortars and calcareous stones), practically insoluble in water, can also be a deterioration product, usually appearing in the form of a surface incrustation.

Qualitative analysis of soluble salts (from a stone or mortar sample) furnishes information about the types of ions (sulfates, chlorides, etc.) present in the sample and gives an indication of the maximum quantity of single ions present. Such information provides some clues as to the type of deterioration in progress and its causes

Origins of the Most Common Salts

(1) Sulfates

The sulfates most commonly found in masonry are hydrated calcium sulfate (gypsum, $CaSO_4 \cdot 2H_2O$) and, more rarely, magnesium sulfate ($MgSO_4$)

The possible origins of such salts are the following:

a) Sulfates are present in agricultural land and can enter a wall through capillary action.

b) Sea water, in addition to chlorides, contains small amounts of sulfates, especially magnesium sulfate. Sea spray can thus deposit sulfate on a surface.

c) Some materials used in the preparation of mortars and plasters can contain small quantities of sulfates as impurities. These can be dissolved in water present in the masonry wall and brought to the surface as efflorescence. Erroneous use of substances like gypsum for restoration can lead to the presence of sulfates.

Ex 16 (continued)

Atmospheric pollution can also produce nitrates. The combustion of hydrocarbons, in addition to creating sulfur dioxide (SO_2), also produces various organic molecules and nitrogen oxides. These nitrogen oxides are extremely dangerous to animal and vegetable life because, in the presence of ultraviolet light, they react with oxygen and the organic molecules present in the polluted atmosphere, forming ozone and organic radicals. The ozone, in turn, oxidizes these radicals to aldehydes and the sulfur dioxide (SO_2) to sulfur trioxide (SO_3). This particular mix of substances produces a dangerous fog called "photochemical smog" which can be found in areas having severe pollution and long periods of strong sunlight (eg Los Angeles or Naples). The nitrogen oxides, through a series of complex reactions, form nitric acid (HNO_3) which, reacting with calcium carbonate ($CaCO_3$), leads to the formation of calcium nitrate ($Ca(NO_3)_2$ $4H_2O$)

(4) Carbonates

Calcium carbonate is a normal constituent of both calcareous stones and of mortars (where it is formed by the carbonization of lime)

Unlike the other salts of efflorescence, calcium carbonate is practically insoluble in water. It can, however, be dissolved as bicarbonate when the water contains a high enough quantity of carbon dioxide (CO_2)

Carbon dioxide is a gas that is normally present in the atmosphere. Its concentration can increase, however, under particular conditions - such as in the case of certain industrial activity or when a large number of people occupy a closed room. If dissolved in water present in a humid wall, carbon dioxide forms carbonic acid (H_2CO_3) which reacts with calcium carbonate ($CaCO_3$), forming the more soluble bicarbonate.

There is thus an equilibrium between these various substances (carbonates, CO_2, water, bicarbonates) which leads to the production of soluble bicarbonates when there is a high concentration of carbon dioxide (CO_2).

When a wall begins to dry, bicarbonate salts in solution come to the surface. As evaporation takes place, the previously established equilibrium shifts in favor of the formation of calcium carbonate which, being practically insoluble, is rapidly deposited on the surface.

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Ex 16 (continued)

SULFUR DIOXIDE (SO_2)

In the air + oxygen (O_2).   on the wall + oxygen (O_2)
UV light and catalysts   and catalysts

Sulfur trioxide (SO_3) +   Sulfur trioxide (SO_3) +
water   water

Sulfuric acid (H_2SO_4)   Sulfuric acid (H_2SO_4) +
+ Ammonia (NH_3) or   Calcium carbonate ($CaCO_3$)
Calcium carbonate ($CaCO_3$)

Ammonium sulfate ((NH_4)$_2SO_4$)   Calcium sulfate ($CaSO_4$)
Calcium sulfate ($CaSO_4$)

(2) Chlorides

These salts (especially sodium chloride ($NaCl$) and calcium chloride ($CaCl_2$)) are principally deposited on a wall by sea spray.

Chlorides can also be the result of impurities in the materials (particularly the sand) used to prepare mortars and plasters

Finally, some types of industrial activity, like the combustion of certain kinds of pit-coal, can result in the presence of gaseous hydrochloric acid (hydrogen chloride, anhydrous (HCl)) in the atmosphere

(1) Nitrites and Nitrates

Nitrites are not often found in walls because they oxidize rapidly into nitrates.

The decomposition of nitrogenous organic material produces nitrites. Thus, nitrites might be present where there is infiltration of sewage water or in the vicinity of old burial sites

In general, however, nitrates are much more frequent. They can have the same origin as nitrites, but can also come from the earth in agricultural regions.

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Ex. 16 (continued)

- For the best possible control of experimental results, it is necessary to execute, simultaneously, so-called "white" reactions. That is, for each of the following tests, the analysis of the actual solution should be followed by an analysis using only the water (the same water used for the preparation of the solutions). The results of the "white" reactions can then be compared with those obtained using the sample solution.

6. Analysis of Sulfates ($SO_4^=$)

- Use one of the solutions in the test tubes

- Add 1 or 2 drops of dilute *hydrochloric acid* (HCl 2N) and 1 or 2 drops of a 10% solution of *barium chloride* ($BaCl_2$).

- A white precipitate of *barium sulfate* ($BaSO_4$), insoluble in dilute nitric acid, indicates the presence of sulfates

- The reaction can be summarized as follows:

$$(SO_4^=) + BaCl_2 \longrightarrow BaSO_4\downarrow + 2Cl^-$$
Sulfate in   White
Solution   Precipitate

7. Analysis of Chlorides (Cl^-)

- Use the solution in the second test tube

- Add 1 or 2 drops of dilute *nitric acid* (HNO_3 2N) and 1 or 2 drops of a solution (0.1N) of *silver nitrate* ($AgNO_3$).

- A whitish-blue, gelatinous precipitate of *silver chloride* (AgCl) indicates the presence of chloride

- The reaction can be summarized as:

$$Cl^- + AgNO_3 \longrightarrow AgCl\downarrow + NO_3^-$$
Chloride in   White
Solution   Precipitate

8. Analysis of Nitrites (NO_2^-)

- Use the solution in the third test tube

- Add 1 or 2 drops of dilute *acetic acid* (CH_3COOH 2N) and 1 or 2 drops of *Griess-Ilosvay's reagent*

- A more or less intense pink color indicates the presence of nitrites

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Ex. 16 (continued)

(5) NOTE: It should be remembered that cement can contain several soluble alkaline salts besides sulfates, nitrites, and nitrates, which are added to give the final product particularly desired characteristics. Thus, if cement has been used in a building where there is some humidity in the walls, the soluble salts present in the cement can migrate toward the original plasters or mortars, causing destructive efflorescence or crystallization upon evaporation.

EQUIPMENT

Mortar and pestle, test tubes, funnels, filter paper

CHEMICALS

- Dilute hydrochloric acid (2N)
- Dilute nitric acid (2N)
- Dilute acetic acid (2N)
- Sulfamic acid
- Barium chloride (10% solution in water)
- Solution of silver nitrate (0.1N)
- Zinc powder
- Griess-Ilosvay's reagent

PROCEDURE

1. Grind the sample to a fine homogeneous powder in a small mortar and pestle; a few milligrams of sample are sufficient for qualitative analysis.

2. Put half of the sample so obtained in a 10 cc test tube for the following analyses and conserve the rest for an eventual control.

3. Add about 2 cc of distilled or deionized water to the test tube and shake gently to dissolve the material.

4. Wait a few minutes until the insoluble part of the sample is deposited at the bottom of the test tube. The solution must be clear; otherwise, it is necessary to filter it using fine filter paper and a small funnel.

5. Conserve the test tube containing the insoluble part of the sample for the analysis of carbonates. Split the clear solution into 4 equal parts, putting each part in a small test tube.

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Ex. 16 (continued)

9. Analysis of Nitrates (NO_3^-)

(a) **Absence of nitrites:** If there are no nitrites (i.e. if the previous reaction was negative), add to the same solution a small quantity of *zinc powder*. The zinc, in the presence of acetic acid, will reduce the nitrates (if present) to nitrites. These will then react with the Griess-Ilosvay's reagent. In this case, therefore, a more or less intense pink color indicates the presence of nitrates.

(b) **Presence of nitrites:** If nitrites were present in the solution utilized for step B, use the solution in the fourth test tube for this analysis. Add a small quantity (one or two crystals) of *sulfamic acid* (HSO_3NH_2) in order to destroy the nitrites. (Use the method employed in Step B on a small part of the solution to be sure that all nitrites are destroyed. If not, continue to add small quantities of sulfamic acid until they are no longer present. Avoid adding an excessive amount of sulfamic acid to the solution.)

- Having eliminated the nitrites, add to the solution 1 or 2 drops of dilute *acetic acid* (CH_3COOH 2N) and 1 or 2 drops of *Griess-Ilosvay's reagent*. Now the solution will not turn pink because the nitrites have been destroyed.

- Add a small amount of *zinc powder*. A more or less intense pink color indicates the presence of nitrates.

10. Analysis of Carbonates ($CO_3^=$)

- Use the insoluble part of the original sample remaining at the bottom of the large test tube.

- Add 1 or 2 drops of concentrated *hydrochloric acid* (HCl).

- Bubbles of gas (CO_2) in the solution indicate the presence of carbonates ($CO_3^=$).

- The reaction can be summarized as follows:

$$CaCO_3 + 2HCl \longrightarrow CaCl_2 + H_2O + CO_2 \uparrow (gas)$$
insoluble   soluble

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Ex. 16 (continued)

11. Expression of Results

The current method of expressing results is to indicate the presence of an ion with a cross (+) and its absence with a minus (-). The relative abundance of an ion is usually indicated by a proportionate number of crosses (++, +++, etc.). The following example should clarify this usage:

SAMPLE	Sulfates	Chlorides	Nitrites	Nitrates	Carbonates
1	++	+	-	+	+
2	+++	+	-	+	+
3	etc.				

-   absence of the ion

+   concentration of the ion at the limit of perceptibility

++   presence of the ion

+++   presence of the ion in notable quantity

+++   presence of the ion as a principal component

DISCUSSION

Using the example above as a model, record your experimental results in the Data Sheet.

Can you make any hypotheses about the origins of the various salts discovered in analysis?

What other analyses might you carry out to verify your hypotheses?

BIBLIOGRAPHY

Istituto Centrale del Restauro. Corso sulle manutenzione di dipinti murali, mosaici, stucchi (DIMOS). Part II, Modulo 3. Rome: Istituto Centrale del Restauro. 1979.

Ex. 16 (continued)
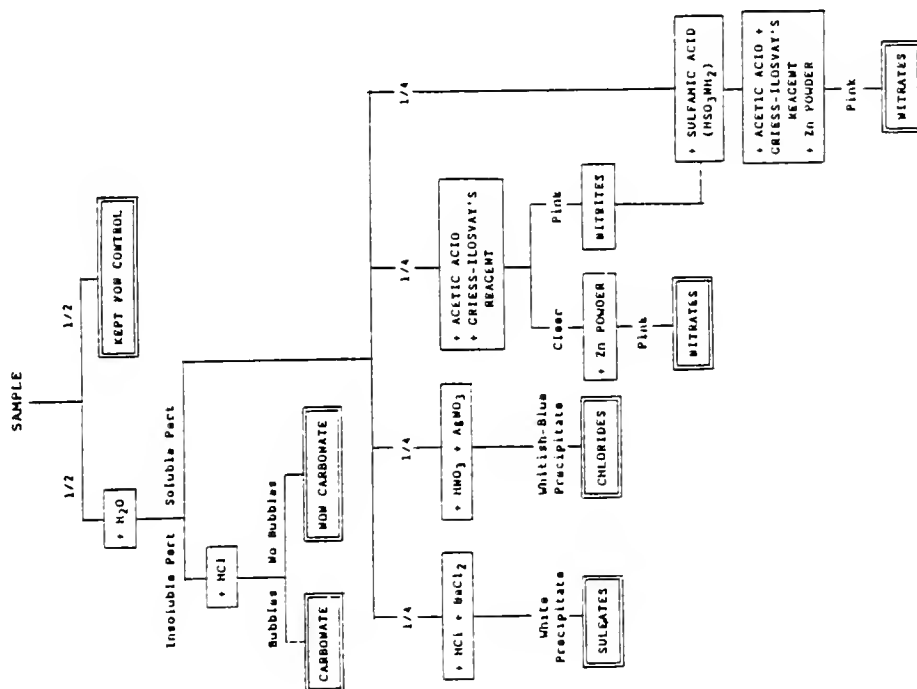
Ex. 16 (continued)

DATA SHEET

SAMPLE	Sulfates	Chlorides	Nitrites	Nitrates	Carbonates

Appendix D

Data Tables and Notes

D.1   Sample Measurements

D.2   Immersion Cycling Times and Solution pH

D.3   Capillary Absorption/Total Immersion Data

D.4   Drying Rate Data

D.5   Sample C

Sample	Depth	Width	Height	Volume	Mass, Dry		Sample	Depth	Width	Height	Volume	Mass, Dry
A	5.275	4 550	4 950		300 30		J	4 740	5 050	5 060		304.38
	5.000	4 545	5.265					4 630	5 095	4 880		
	5.255	4 320	4 880					4 780	5 120	4 970		
	4 925	4 395	5 245					4 550	5 085	4 840		
Mean	5.114	4.453	5.085	115.780			Mean	4.675	5.088	4.938	117.434	
B	5 015	4 445	5.160		307 06		K	5 090	4 920	4 870		327 43
	5 150	4 290	5.155					4 780	4 845	5 020		
	5 180	4 395	5.190					5 050	5 500	4 920		
	5.175	4 675	5.150					4 825	5 590	5 000		
Mean	5.130	4.451	5.164	117.914			Mean	4.936	5.214	4.953	127.459	
C	5 100	4 700	5 780		363 00		L	5 025	4 915	4 215		259 90
	5 130	4 870	5 095					3 995	4 945	5 070		
	5 085	4 880	5 800					5 050	4 945	4 160		
	5 670	4 700	5.080					3 855	4 875	5 120		
Mean	5.246	4.788	5.439	136.602			Mean	4.481	4.920	4.641	102.329	
D	5 095	4 845	4 855		313.17		M	4 885	4 845	3 825		235 87
	4 845	4 885	5 080					4 145	4 850	4 860		
	5 035	4 930	4 930					4 950	4 860	3.665		
	4 930	4 895	5 050					3 855	4 880	4 795		
Mean	4.976	4.889	4.979	121.121			Mean	4.459	4.859	4.286	92.857	
E	5.660	4 700	5.055		361.82		N	4 940	4 040	4 790		269 30
	5 225	4 610	5 825					4 725	4 050	4 890		
	5 815	4 620	5.185					4 915	4 785	4 870		
	5 369	4 755	5 225					4 785	4 840	4 865		
Mean	5.517	4.671	5.323	137.174			Mean	4.841	4.429	4.854	104.068	
F	4 970	4 915	4 275		264 45		O	4 800	5 590	4 650		306 34
	4 420	4 900	4 765					4 400	5 090	4 895		
	5 040	4 910	4 130					4 800	5.150	4 730		
	4 275	4 920	4 720					4 495	5 530	4 925		
Mean	4.676	4.911	4.473	102.716			Mean	4.624	5.340	4.800	118.516	
G	4 750	4 020	4 980		252 73		P	4 750	4 360	4.260		214 36
	4 930	4 225	4 200					4 100	4 400	4 795		
	4 540	4 340	5 015					4 625	4 095	4 100		
	4 955	4 120	4 945					3 970	3 975	4 895		
Mean	4.794	4.176	4.785	95.795			Mean	4.361	4.208	4.513	82.804	
H	5 050	4 615	5 065		299 53		Q	5 000	2 455	4 725		206 76
	4 990	4 660	4 995					4 660	2.580	4 695		
	5 000	4 655	5 000					5 000	4 740	4 765		
	4 955	4 525	5 060					4 650	4 595	4 735		
Mean	4.999	4.614	5.030	116.007			Mean	4.828	3.593	4.730	82.031	
I	4 715	4 455	5 065		264 89							
	5 100	4 290	4 670									
	4 735	4 215	4 990									
	5 025	4 480	4 615									
Mean	4.894	4.360	4.835	103.163								

Immersion Cycling Times and pH						
Date	**Time Out**	**Time In**	**pH Lime**	**pH Gypsum**	**pH Salt**	**Notes**
3/5/99		18:00	12.44		8.94	Gypsum not available
3/6/99	9:00	20:30	13.08	8.95	8.86	Trouble calibrating pH meter
3/7/99	10:00	19:30	13.03	6.30	8.70	
3/8/99	7:20	16:15	12.99	7.03	8.67	
3/15/99	9:50	17:45	12.35	7.37	8.23	Before solution change
			11.96	7.53	8.41	After solution change
3/16/99	9:00	18:45	12.33	7.51	8.37	
3/17/99	9:55	19:30	12.14	7.23	8.18	
3/18/99	11:30	22:45	12.06	7.47	8.25, 7.93	
3/19/99	11:30	18:00	11.93, 12.00	7.34, 7.12	8.34, 7.87	
3/20/99	12:10	18:45	11.76, 11.80	7.36, 7.11	8.23, 7.80	
3/21/99	10:30	20:45	11.85, 12.11	7.77, 7.18	8.24, 7.85	
			12.5	6.3	6.5	Began measuring pH with paper
3/22/99	11:10	20:35	12.5	6.5	8.0	New solutions
3/23/99	11:30	19:00	12.5	6.3	7.8	
3/24/99	13:00	19:30	12.5	6.3	7.4, 7.5	
3/25/99	10:00	22:00	12.5	5.5, 6.0	7.0, 7.1	
3/26/99	13:00	22:00	12.5	6.3	6.8, 6.9	
3/27/99	14:00	2:00	12.5	6.3	6.9	
3/28/99	15:00	22:00	12.5	6.3	6.7	
3/29/99	14:30	20:30	12.5	6.3	6.5, 6.6	New solutions
3/31/99	12:00	19:00	12.5	6.3	6.3, 6.5	
4/1/99	12:30	19:30	12.5	6.3	6.3, 6.5	
4/2/99	10:50	18:00	12.5	6.3	6.3, 6.5	Immersed to saturate for
						Drying Index
4/5/99	Weight, saturated		H: 301.80	A: 302.15	F: 266.43	
			O: 309.20	D: 315.47	J: 306.39	
4/7/99	Weight, saturated		H: 301.83	A: 302.16	F: 266.44	
			O: 309.24	D: 315.50	J: 306.41	
	Final pH		12.5	5.5, 6.0	6	pH Evap. Gypsum: 5.5, 6.0

Capillary Water Absorption and Capillary Absorption Coefficient							
NORMAL 11/85							
Sample E, with foliation							
Dry Weight (g):	361.85						
Surface Area (cm³):	27.170						
Date/Time	Elapsed	Elapsed	Weight (g)	WH20 (g)	dWH20 (%)	dWdry(%)	Mi(g/cm²)
	Time(min)	Time(hrs)					
3/21/99 10:25	0	0.00	361.85	0.00	0.00%	0.00%	0.000	
3/21/99 10:29	4	0.07	362.28	0.43	100.00%	0.12%	0.016	No blot
3/21/99 10:34	9	0.15	362.19	0.34	-26.47%	0.09%	0.013	
3/21/99 10:39	14	0.23	362.24	0.39	12.82%	0.11%	0.014	
3/21/99 10:55	30	0.50	362.45	0.60	35.00%	0.17%	0.022	
3/21/99 11:10	45	0.75	362.54	0.69	13.04%	0.19%	0.025	
3/21/99 11:25	60	1.00	362.66	0.81	14.81%	0.22%	0.030	
3/21/99 12:25	120	2.00	363.16	1.31	38.17%	0.36%	0.048	
3/21/99 13:26	181	3.02	363.61	1.76	25.57%	0.49%	0.065	
3/21/99 14:26	241	4.02	363.97	2.12	16.98%	0.59%	0.078	
3/21/99 18:36	491	8.18	364.62	2.77	23.47%	0.77%	0.102	
3/21/99 22:31	726	12.10	364.64	2.79	0.72%	0.77%	0.103	
3/22/99 11:10	1485	24.75	364.63	2.78	-0.36%	0.77%	0.102	
3/23/99 11:30	2945	49.08	364.75	2.90	4.14%	0.80%	0.107	
3/24/99 13:00	4475	74.58	364.76	2.91	0.34%	0.80%	0.107	
3/25/99 20:10	6345	105.75	364.83	2.98	2.35%	0.82%	0.110	
3/26/99 13:00	7355	122.58	364.86	3.01	1.00%	0.83%	0.111	
3/27/99 14:00	8855	147.58	364.90	3.05	1.31%	0.84%	0.112	
3/28/99 15:00	10295	171.58	364.92	3.07	0.65%	0.85%	0.113	

Capillary Water Absorption
Sample E: With Foliation

Capillary Water Absorption and Capillary Absorption Coefficient						
NORMAL 11/85						
Sample K, against foliation						
Dry Weight (g):	327.46					
Surface Area (cm^3):	25.705					
Date/Time	Elapsed Time(min)	Weight (g)	WH20 (g)	dWH20 (%)	dWdry(%)	Mi(g/cm^2)
3/21/99 10:25	0	327.46	0.00	0.00%	0.00%	0.000
3/21/99 10:31	6	328.04	0.58	100.00%	0.18%	0.023
3/21/99 10:35	10	328.18	0.72	19 44%	0.22%	0.028
3/21/99 10:40	15	328.34	0.88	18.18%	0.27%	0.034
3/21/99 10:56	31	328.71	1.25	29.60%	0.38%	0.049
3/21/99 11:10	45	328.84	1.38	9.42%	0.42%	0.054
3/21/99 11:25	60	328.97	1.51	8.61%	0.46%	0.059
3/21/99 12:25	120	329.54	2.08	27.40%	0.64%	0.081
3/21/99 13:28	183	329.79	2.33	10.73%	0.71%	0.091
3/21/99 14:28	243	329.85	2.39	2.51%	0.73%	0.093
3/21/99 18:36	491	329.90	2.44	2.05%	0.75%	0.095
3/21/99 22:31	726	329.92	2.46	0.81%	0.75%	0.096
3/22/99 11:09	1484	329.90	2.44	-0.82%	0.75%	0.095
3/23/99 11:30	2945	330.06	2.60	6.15%	0.79%	0.101
3/24/99 13:00	4475	330.09	2.63	1.14%	0.80%	0.102
3/25/99 20:10	6345	330.16	2.70	2.59%	0.82%	0.105
3/26/99 13:00	7355	330.17	2.71	0.37%	0.83%	0.105
3/27/99 14:00	8855	330.22	2.76	1.81%	0.84%	0.107
3/28/99 15:00	10295	330.22	2.76	0 00%	0.84%	0.107

Capillary Water Absorption
Sample K: Against Foliation

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Capillary Water Absorption and Capillary Absorption Coefficient							
NORMAL 11/85							
Sample P, with foliation							
Dry Weight (g):	214.39						
Surface Area (cm³):	20.284						
Date/Time	Elapsed Time(min)	Elapsed Time(hrs)	Weight (g)	WH20 (g)	dWH20 (%)	dWdry(%)	$Mi(g/cm^2)$
3/21/99 10:25	0	0.00	214.39	0.00	0.00%	0.00%	0.000
3/21/99 10:30	5	0.08	214.82	0.43	100.00%	0.20%	0.021
3/21/99 10:35	10	0.17	214.96	0.57	24.56%	0.27%	0.028
3/21/99 10:40	15	0.25	215.10	0.71	19.72%	0.33%	0.035
3/21/99 10:55	30	0.50	215.41	1.02	30.39%	0.48%	0.050
3/21/99 11:10	45	0.75	215.54	1.15	11.30%	0.54%	0.057
3/21/99 11:25	60	1.00	215.68	1.29	10.85%	0.60%	0.064
3/21/99 12:25	120	2.00	215.91	1.52	15.13%	0.71%	0.075
3/21/99 13:27	182	3.03	215.93	1.54	1.30%	0.72%	0.076
3/21/99 14:26	241	4.02	215.98	1.59	3.14%	0.74%	0.078
3/21/99 18:36	491	8.18	216.00	1.61	1.24%	0.75%	0.079
3/21/99 22:31	726	12.10	216.01	1.62	0.62%	0.76%	0.080
3/22/99 11:10	1485	24.75	215.97	1.58	-2.53%	0.74%	0.078
3/23/99 11:30	2945	49 08	216.09	1.70	7.06%	0.79%	0.084
3/24/99 13:00	4475	74.58	216.10	1.71	0.58%	0.80%	0.084
3/25/99 20:10	6345	105.75	216.15	1.76	2.84%	0.82%	0.087
3/26/99 13:00	7355	122.58	216.16	1.77	0.56%	0.83%	0.087
3/27/99 14:00	8855	147.58	216.20	1.81	2.21%	0.84%	0.089
3/28/99 15:00	10295	171.58	216.20	1.81	0.00%	0.84%	0.089

Capillary Water Absorption
Sample P: With Foliation

Sample A	ptw	300.3								Sample A
Fin dry wt	300.24		Treatment	Gypsum Soak						
Date	Time	Elapsed	Weight	formula	%dry					Sample A
		Time							Elapsed Tim	Qi (%)
4/7/99	12:55	0.00	302.16						0.00	0.62
	13:25	0.50	301.73	0.00	0.14				0.50	0.48
	14:00	1.08	301.38	0.55	0.12				1.08	0.36
	14:30	1.58	301.28	0.89	0.03				1.58	0.33
	15:05	2.17	301.19	0.91	0.03				2.17	0.30
	15:30	2.58	301.11	0.92	0.03				2.58	0.27
	16:10	3.25	301.10	0.99	0.00				3.25	0.27
	17:05	4.17	301.03	0.94	0.02				4.17	0.24
	18:10	5.25	300.97	0.95	0.02				5.25	0.22
	20:40	7.75	300.87	0.92	0.03				7.75	0.19
4/8/99	13:15	24.33	300.59	0.82	0.09				24.33	0.10
4/9/99	12:50	47.92	300.52	0.96	0.02				47.92	0.07
4/10/99	14:30	73.58	300.44	0.95	0.03				73.58	0.05

Drying Curve
Sample A: Gypsum Soak

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Sample B	ptw	307 06								
Fin dry wt	307 20		Treatment	Gypsum Evaporation						
Date	Time	Elapsed	Weight	formula	%dry					Sample B
		Time							Elapsed Tim	Qi (%)
4/7/99	13.25	0	309.51						0.00	0 80
	14 05	0.5	308.96	0.00	0.18				0.50	0 62
	14 30	1 08	308 62	0.62	0.11				1 08	0 51
	15 05	1.67	308 43	0 82	0.06				1.67	0 45
	15.35	2.17	308.33	0 92	0.03				2.17	0 41
	16.15	2 83	308 23	0 92	0.03				2.83	0.38
	17 05	3 67	308 15	0 94	0 03				3 67	0.35
	18.10	4.75	308 07	0 94	0.03				4 75	0.33
	20 40	7.25	307.94	0.92	0 04				7 25	0.29
4/8/99	13 15	23 83	307.61	0.83	0.11				23 83	0.18
4/9/99	12:50	47 41	307.50	0.95	0.04				47.41	0.14
4/10/99	14.30	73 17	307.41	0.96	0 03				73 17	0 11

Drying Curve
Sample B: Gypsum Evaporation

Sample D	ptw	313.17							
Fin dry wt	313.14		Treatment	Gypsum Soak					
Date	Time	Elapsed Time	Weight	formula	%dry			Elapsed Tim	Sample D Qı (%)
4/7/99	12:55	0.00	315.50					0.00	0 74
	13:25	0.50	315.04	0.00	0.15			0.50	0.60
	14:00	1 08	314.44	0.43	0.19			1.08	0.41
	14.30	1.58	314.25	0.77	0.06			1.58	0.34
	15:05	2.17	314.13	0.91	0.04			2.17	0.31
	15.30	2.58	314.06	0.95	0.02			2.58	0.28
	16:10	3.25	314 00	0 96	0.02			3.25	0.27
	17.05	4 17	313.92	0.94	0.03			4 17	0 24
	18 10	5.25	313.85	0.96	0.02			5.25	0.22
	20 40	7 75	313 74	0 94	0 04			7 75	0 18
4/8/99	13 15	24 33	313 44	0 85	0 10			24 33	0 09
4/9/99	12.50	47 92	313 35	0 96	0 03			47 92	0 06
4/10/99	14 30	73.58	313.28	0 97	0.02			73.58	0 04

Drying Curve
Sample D: Gypsum Soak

Sample E	ptw	361.85							
Dry Wt.	361.69		Treatment.	None					
Date	Time	Elapsed	Weight	formula	%dry				Sample E
		Time						Elapsed Tim	Qi (%)
3/31/99	12:10	0	364.92					0.00	0.85
	12:55		364.14	0.00	0.22			0.75	0.63
	13:40		363.52	0.56	0.17			1.50	0.46
	14:25		363.28	0.85	0.07			2.25	0.40
	15:25		363.12	0.91	0.04			3.25	0.35
	16:25		362.98	0.93	0.04			4.25	0.31
	18:50		362.81	0.92	0.05			6.67	0.27
4/1/99	12:30		362.27	0.80	0.15			24.33	0.12
	19:40		362.23	0.99	0.01			31.50	0.11
4/2/99	10:55		362.11	0.96	0.03			46.75	0.07
4/5/99	14:30		361.90	0.93	0.06			122.33	0.01

Drying Curve
Sample E: Untreated

Sample F	ptx	264.45							
Fin dry wt:	264.63		Treatment:	Salt Soak					
Date	Time	Elapsed	Weight	formula	%dry				Sample F
		Time						Elapsed Tin	Qi (%)
4/7/99	12:55	0	266.44					0.00	0.75
	13:25	0.50	266.24	0.00	0.08			0.50	0.68
	14:00	1.08	265.82	0.32	0.16			1.08	0.52
	14:30	1.58	265.67	0.81	0.06			1.58	0.46
	15:05	2.17	265.59	0.91	0.03			2.17	0.43
	15:30	2.58	265.53	0.93	0.02			2.58	0.41
	16:10	3.25	265.47	0.94	0.02			3.25	0.39
	17:05	4.17	265.39	0.92	0.03			4.17	0.36
	18:10	5.25	265.35	0.96	0.02			5.25	0.34
	20:40	7.75	265.25	0.92	0.04			7.75	0.30
4/8/99	13:15	24.33	264.99	0.82	0.10			24.33	0.20
4/9/99	12:50	47.92	264.88	0.93	0.04			47.92	0.16
4/10/99	14:30	73.58	264.80	0.95	0.03			73.58	0.13

Drying Curve
Sample F: Salt/Gypsum Soak

Sample H	ptx	299.53							
Fin dry wt	299 73		Treatment	Lime Soak					
Date	Time	Elapsed Time	Weight	formula	%dry			Elapsed Tim	Sample H Qi (%)
4/7/99	12:55	0 00	301 83					0 00	0.77
	13.25	0.50	301 66	0.00	0.06			0.50	0.71
	14:00	1.08	301.56	0.63	0.03			1 08	0 68
	14.30	1.58	301 49	0.79	0.02			1.58	0.65
	15:05	2.17	301.44	0.87	0.02			2.17	0 64
	15:30	2.58	301.40	0.91	0.01			2.58	0.62
	16:10	3.25	301.34	0.88	0.02			3.25	0.60
	17:05	4.17	301.25	0.84	0.03			4.17	0.57
	18:10	5.25	301.15	0.85	0.03			5.25	0 54
	20:40	7.75	300.95	0.77	0.07			7.75	0.47
4/8/99	13:15	24.33	300.42	0.62	0.18			24.33	0.30
4/9/99	12:50	47.92	300.21	0.87	0.07			47.92	0.23
4/10/99	14.30	73.58	300.06	0.92	0.05			73.58	0 18

Drying Curve
Sample H: Lime Soak

Sample 1	ptw	264.89								
Fin Dry wt.	264 96		Treatment.	Gypsum Evaporation						
Date	Time	Elapsed	Weight	formula	%dry				Sample 1	
		Time						Elapsed Tim	Qı (%)	
4/7/99	13:25	0	267.61					0.00	1.03	-
	14.05	0.5	267.01	0.00	0.23			0.50	0.80	
	14:30	1.08	266.52	0.55	0.18			1.08	0.62	
	15:05	1.67	266.18	0.76	0.13			1.67	0.49	
	15:35	2.17	266.07	0.93	0.04			2.17	0.45	
	16:15	2.83	265.97	0.94	0.04			2.83	0.41	
	17:05	3.67	265.86	0.94	0.04			3.67	0.37	
	18:10	4.75	265.78	0.96	0.03			4.75	0.34	
	20:40	7.25	265.65	0.93	0.05			7.25	0.29	
4/8/99	13 15	23.83	265 30	0 85	0 13			23.83	0 15	
4/9/99	12 50	47 41	265.19	0 95	0.04			47 41	0.11	
4/10/99	14.30	73.17	265.11	0 97	0 03			73.17	0.08	

Drying Curve
Sample I: Gypsum Evaporation

Sample J	ptw	304.38								
Fin dry wt	304.55		Treatment	Salt Soak						
Date	Time	Elapsed	Weight	formula	%dry					Sample J
		Time							Elapsed Tim	Qi (%)
4/7/99	12:55	0	306.41						0.00	0.67
	13:25	0.50	306.22	0.00	0.06				0.50	0.60
	14:00	1.08	305.77	0.30	0.15				1.08	0.46
	14:30	1.58	305.64	0.83	0.04				1.58	0.41
	15:05	2.17	305.55	0.90	0.03				2.17	0.38
	15:30	2.58	305.50	0.95	0.02				2.58	0.37
	16:10	3.25	305.43	0.93	0.02				3.25	0.34
	17:05	4.17	305.37	0.94	0.02				4.17	0.33
	18:10	5.25	305.30	0.94	0.02				5.25	0.30
	20:40	7.75	305.22	0.93	0.03				7.75	0.28
4/8/99	13:15	24.33	304.93	0.80	0.10				24.33	0.18
4/9/99	12:50	47.92	304.84	0.94	0.03				47.92	0.15
4/10/99	14:30	73.58	304.75	0.95	0.03				73.58	0.12

Drying Curve
Sample J: Salt/Gypsum Soak

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Sample K	ptw	327 46							
Dry Wt	327.34		Treatment	None					
Date	Time	Elapsed Time	Weight	formula	%dry			Elapsed Tim	Sample K Qi (%)
3/31/99	12.10	0	330.27					0.00	0.86
	12:55		329.55	0.00	0.22			0 75	0 64
	13 40		328.79	0 49	0.23			1.50	0 41
	14.25		328.52	0 85	0.08			2.25	0.32
	15:25		328.37	0.92	0.05			3.25	0.28
	16.25		328.27	0.95	0.03			4.25	0.25
	18:50		328.12	0.93	0.05			6.67	0.20
4/1/99	12:30		327.70	0.84	0.13			24.33	0.07
	19:40		327.67	0.99	0.01			31.50	0.06
4/2/99	10:55		327.58	0.97	0.03			46 75	0 04
4/5/99	14 30		327 46	0.96	0.04			122.33	0.00

Drying Curve
Sample K: Untreated

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Sample O	ptx	306 34								Sample O
Fin dry wt	306 67		Treatment	Lime Soak						
Date	Time	Elapsed Time	Weight	formula	%dry				Elapsed Tim	Sample O Qı (%)
4/7/99	12:55	0.00	309.24						0.00	0.95
	13:25	0.50	308.86	0.00	0.12				0.50	0 82
	14:00	1.08	308.56	0.56	0 10				1.08	0 72
	14.30	1.58	308.45	0.86	0.04				1.58	0.69
	15:05	2 17	308.29	0.83	0 05				2 17	0.64
	15.30	2.58	308.19	0.90	0.03				2.58	0.60
	16·10	3 25	308.08	0 91	0 04				3.25	0 57
	17:05	4 17	307.92	0.88	0 05				4 17	0 52
	18 10	5.25	307 78	0.90	0 05				5.25	0 47
	20 40	7 75	307 55	0.86	0 07				7.75	0.39
4/8/99	13 15	24 33	307 11	0.79	0.14				24.33	0.25
4/9/99	12.50	47 92	306.97	0.94	0 05				47 92	0.21
4/10/99	14.30	73.58	306 85	0.95	0 04				73.58	0 17

Drying Curve
Sample O: Lime Soak

Sample P	ptw	214.39							
Dry Wt.	214.32		Treatment	None					
Date	Time	Elapsed	Weight	formula	%dry				Sample P
		Time						Elapsed Tim	Qı (%)
3/31/99	12:10	0	216.20					0 00	0 84
	12:55		215.60	0.00	0 28			0.75	0.56
	13 40		215.16	0.58	0.21			1.50	0.36
	14.25		215.03	0 89	0 06			2.25	0 30
	15.25		214 94	0 93	0 04			3 25	0 26
	16.25		214.86	0 94	0 04			4.25	0 22
	18.50		214 76	0.93	0 05			6.67	0 17
4/1/99	12:30		214 52	0.86	0 11			24.33	0 06
	19 40		214.50	0 99	0.01			31.50	0 05
4/2/99	10:55		214.45	0 97	0 02			46.75	0 03
4/5/99	14.30		214.39	0.97	0.03			122.33	0 00

Drying Curve
Sample P: Untreated

Capillary Water Absorption and Capillary Absorption Coefficient							
NORMAL 11/85							
Sample C, against foliation							
Dry Weight (g):	363.04						
Surface Area (cm³):	27.628						
Date/Time	Elapsed	Elapsed	Weight (g)	WH20 (g)	dWH20 (%)	dWdry(%)	Mi(g/cm²)
	Time(min)	Time(hrs)					
3/21/99 10:25	0	0.00	363.04	0.00	0.00%	0.00%	0.000
3/21/99 10:30	5	0.08	363.17	0.13	100.00%	0.04%	0.005
3/21/99 10:35	10	0.17	363.23	0.19	31.58%	0.05%	0.007
3/21/99 10:40	15	0.25	363.28	0.24	20.83%	0.07%	0.009
3/21/99 10:56	31	0.52	363.42	0.38	36.84%	0.10%	0.014
3/21/99 11:10	45	0.75	363.44	0.40	5.00%	0.11%	0.014
3/21/99 11:25	60	1.00	363.48	0.44	9.09%	0.12%	0.016
3/21/99 12:25	120	2.00	363.60	0.56	21.43%	0.15%	0.020
3/21/99 13:27	182	3.03	363.78	0.74	24.32%	0.20%	0.027
3/21/99 14:27	242	4.03	363.92	0.88	15.91%	0.24%	0.032
3/21/99 18:36	491	8.18	364.42	1.38	36.23%	0.38%	0.050
3/21/99 22:31	726	12.10	364.71	1.67	17.37%	0.46%	0.060
3/22/99 11:09	1484	24.73	365.40	2.36	29.24%	0.65%	0.085
3/23/99 11:30	2945	49.08	365.60	2.56	7.81%	0.71%	0.093
3/24/99 13:00	4475	74.58	365.61	2.57	0.39%	0.71%	0.093
3/25/99 20:10	6345	105.75	365.65	2.61	1.53%	0.72%	0.094
3/26/99 13:00	7355	122.58	365.66	2.62	0.38%	0.72%	0.095
3/27/99 14:00	8855	147.58	365.69	2.65	1.13%	0.73%	0.096
3/28/99 15:00	10295	171.58	365.69	2.65	0.00%	0.73%	0.096

Capillary Water Absorption
Sample C: Against Foliation

Sample C	ptw	363.04								
Dry Wt:	362.92		Treatment:	None						
Date	Time	Elapsed	Weight	formula	%dry					
		Time (h)							Elapsed Tin	Qi (%)
3/31/99	12:10	0	365.77						0.00	0.75
	12:55	0.75	365.06	0.00	0.20				0.75	0.56
	13:40	1.5	364.78	0.72	0.08				1.50	0.48
	14:25	2.25	364.63	0.87	0.04				2.25	0.44
	15:25	3.25	364.50	0.90	0.04				3.25	0.40
	16:25	4.25	364.40	0.93	0.03				4.25	0.37
	18:50	6.67	364.25	0.90	0.04				6.67	0.33
4/1/99	12:30	24.33	363.78	0.76	0.13				24.33	0.20
	19:40	31.5	363.74	0.98	0.01				31.50	0.19
4/2/99	10:55	46.75	363.60	0.94	0.04				46.75	0.15
4/5/99	14:30	122.33	363.32	0.89	0.08				122.33	0.08

Drying Curve
Sample C: Untreated
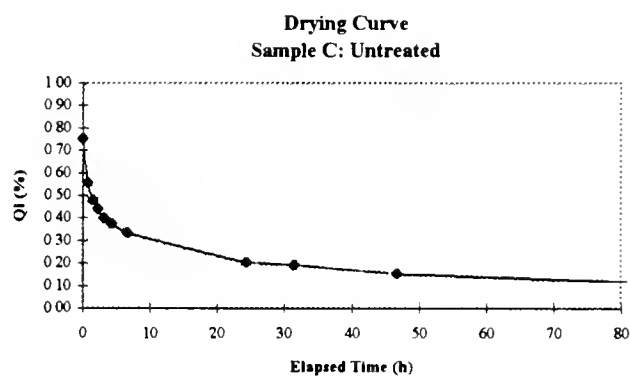
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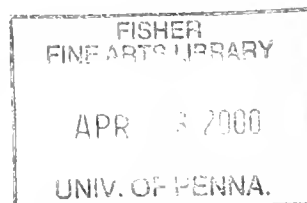
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